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U. S. DEPARTMENT OF AGRICULTURE.

BUREAU OF CHEMISTRY—BULLETIN NO. 65.

H. W. WILEY, Chief.

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## PROVISIONAL METHODS

FOR THE

# ANALYSIS OF FOODS

ADOPTED BY THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS,  
NOVEMBER 14-16, 1901.

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EDITED BY

H. W. WILEY, SECRETARY,

WITH THE COLLABORATION OF

W. D. BIGELOW, REFEREE ON FOOD ADULTERATION.



WASHINGTON:

GOVERNMENT PRINTING OFFICE.

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## LETTER OF TRANSMITTAL

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U. S. DEPARTMENT OF AGRICULTURE,  
BUREAU OF CHEMISTRY,  
*Washington, D. C., January 7, 1902.*

SIR: I have the honor to submit for your inspection and approval the manuscript of the Provisional Methods of the Association of Official Agricultural Chemists for the Analysis of Foods, with the recommendation that it be published as Bulletin No. 65 of the Bureau of Chemistry.

H. W. WILEY,  
*Chief of the Bureau of Chemistry and Secretary of the  
Association of Official Agricultural Chemists.*

Hon. JAMES WILSON,  
*Secretary of Agriculture.*

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## INTRODUCTION.

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At the meeting of the association in 1900 it was decided, at the suggestion of Mr. Kilgore, the retiring president, to divide the subject of food adulteration into a number of general classes, and make a systematic effort to outline methods for their examination. With this in view, the referee in charge of this subject was instructed to secure the cooperation of associate referees, each of whom should prepare methods for the examination of one or more classes of foods. It was recognized that these methods could not all be prepared at once, but it was the desire of the association that a beginning be made, and that the work be prosecuted with as much vigor as possible. The work was immediately organized, and the cooperation of the following associates was secured: W. M. Allen, W. H. Ellis, William Frear, F. T. Harrison, A. E. Leach, J. A. Le Clerc, A. McGill, A. S. Mitchell, L. S. Munson, L. M. Tolman, H. W. Wiley, and A. L. Winton.

The reports, when completed, were forwarded to the referee, printed, and distributed to a mailing list of about 250 chemists for suggestions and criticisms, and a meeting of the entire committee was convened just before the meeting of the association in November, 1901. The methods as amended at this meeting were reported to the association and adopted provisionally. In several cases the reports which follow are the result of extensive work which was performed largely for the preparation of these methods. In other cases it has only been possible to take up a portion of the subject; and in still other instances it was found necessary to defer reports for another year.

On the whole, it may be said that the methods which were presented are more complete than was anticipated. Several who had not expected to make any report until the following year have been able to prepare a creditable outline of their subjects, and all the reports promised for this year have been received. It is considered, however, by those who have the matter in hand, that only a beginning has been made and that experience will indicate numerous changes in the methods which will be advantageous. At the same time the subject is placed in such a position that it may be considered in detail at an earlier date than was expected.

The writer desires to express his obligation to all of his associates, and especially to the local associates, Messrs. Munson and Tolman, for their cordial, prompt, and efficient cooperation.

W. D. BIGELOW,  
*Referee on Food Adulteration.*

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# PROVISIONAL METHODS FOR THE ANALYSIS OF FOODS, ADOPTED BY THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, NOVEMBER 14-16, 1901.

## I. MEAT AND MEAT PRODUCTS.

By W. D. BIGELOW,

*In Charge of Food Laboratory, Bureau of Chemistry, U. S. Department of Agriculture.*

### A. MEAT.

#### 1.—IDENTIFICATION OF SPECIES.

When dealing with large pieces of meat, and especially with fresh meat, the determination of the species of animal from which it was taken is the work of the veterinarian rather than the chemist, although the data obtained by the latter are often conclusive as to the variety of meat present. The physical appearance of the meat, its luster, grain, compactness, the presence or absence of the marbled appearance due to intermuscular fat, the size and shape of the bones, and the color and consistency of the fatty tissue must all be taken into account. Many of these characteristics are destroyed by curing or smoking, and none of them are retained by chopped meat, sausage, potted meat, or other preparations of like nature.

In such cases we must depend mainly on the results of chemical examination. The percentage of glycogen, added to the percentage of reducing sugar, is often of value in detecting horse meat in preparations which are supposed to consist of beef. Certain results obtained in the examination of fat separated from the meat by heat or by extraction with organic solvents also afford valuable data. Among the factors which are of value for this purpose may be mentioned the iodine number, melting point, freezing point, index of refraction, and to a less extent the specific gravity, acetyl number, and Maumené value.<sup>a</sup> The meat from embryonic animals and from animals killed before they are suitable for food may often be detected by its moist, clammy nature and high water content.

#### 2.—EXAMINATION OF POISONOUS MEAT AND OTHER FOODS.<sup>b</sup>

From a hygienic standpoint the recognition of diseased meat is a matter of prime importance. The inspection of fresh meats for the purpose of detecting animal parasites such as trichinæ and vegetable parasites such as the lumpy-jaw fungus, the bacillus of tuberculosis, and other disease-producing bacteria, need not concern the analyst. It is only when a food, presumably wholesome, is found to have poisoned one or more individuals that the analyst must, to some degree at least, distinguish between the agents that may play a causative rôle.

The ordinary foods of man are liable to become poisonous from either of the three causes: (1) Trichinæ in pork, (2) metals, and (3) bacterial products.

<sup>a</sup>See Appendix, p. 149.

<sup>b</sup>The matter under this heading was written for this bulletin by Dr. F. G. Novy, of Ann Arbor, Mich.

## (a) TRICHINÆ.

Pork, and sausage containing pork, which has caused sickness should be examined at once for trichinæ.<sup>a</sup>

## (b) POISONOUS METALS.

The poisonous metals arsenic, antimony, tin, lead, copper, and zinc are to be considered. It should be borne in mind, however, that food poisoning from metals is extremely rare compared with the causes mentioned under bacterial products. It is furthermore not uncommon to find minute amounts of tin or lead in canned meats or other canned food, and the accidental development of toxic properties in such canned goods can not stand in any causal relation to such minimal amount of metals. A single small dose of copper, lead, tin, or other metal need not in itself cause any unpleasant effect, but continued dosing with such small amounts may in the end give rise to disturbances. Several instances of this kind may be mentioned. Vaughan, some years ago, found the cattle in a Western mining region to be dying off because the stream from which they drank received the washings from a hydraulic mining camp. An investigation showed that both arsenic and antimony were present in solution as well as in suspension. Again, a garrison stationed on an East Indian island was obliged to use exclusively water stored up in a galvanized iron tank, with the result that chronic gastroenteritis developed in nearly every man. The experience with arsenic in beer in Manchester is very recent. Several hundred cases of chronic peripheral neuritis followed the continued use of beer containing minute amounts of this poison. On the other hand, acute poisoning from metals added intentionally or by mistake to foods are too well known. The amount of poison in such cases is such as to render the whole matter easy of solution.

From what has been said it is evident that poisonous meats could only under the most exceptional conditions owe this property to metals. A chemical examination, beyond revealing the merest traces of such metals as tin or lead, would mean nothing. More than that, such an examination requires a relatively large amount of material, and it not infrequently happens that the chemist hastens on with his chemical examination, to which he subjects all or nearly all of his material, so that when he attains a negative result scarcely any of the original substance is left for examination along other lines. For the determination of heavy metals, proceed as directed under vegetables (p. 52).

## (c) BACTERIAL PRODUCTS.

The vast majority of all food poisonings are due to the invasion of the food by bacteria. The mere fact that bacteria are present in a meat is not evidence that such food is poisonous. Many, in fact most, of the bacteria which invade food are incapable of producing poisons, but they may grow and multiply and give rise to observable decomposition changes. Such changes can be spoken of as simple decomposition due to invasion by nonpoisonous bacteria. When, however, poison-producing germs develop in the meat, then as food it becomes poisonous. It is a noteworthy fact that a food may be highly poisonous without any visible indication of decomposition. In other words, odor and taste are not always reliable guides as to the innocuousness of a food.

The toxicogenic germ present in a food may be such that it can not grow in the body, and hence it is obvious that the poisonous effects which are observed are due to the poison which the germ had elaborated while growing in the meat. Such poisonings are pure intoxications. Another type of germ not only grows in the meat where it makes some poison, but it can also grow in the body, and as a result it continues to elaborate such poison for some time after being ingested. And, lastly, a germ may be present which can not grow at ordinary temperature, but does grow in the

<sup>a</sup> Fischöder, Leitfaden der praktischen Fleischschau; Ostertag, Handbuch der Fleischschau; Walley, A Practical Guide to Meat Inspection.



body, in which case the poisoning is a true infection. Instances are known where diseases of animals have been transmitted by eating the flesh.

In view of the fact that bacteria are, above all, the most common cause of poisonous meat, it follows that the examination should primarily be made from that standpoint. To sum up what has already been said: Given a poisonous meat, the first procedure is to detect or exclude the presence of trichinae. If they are not found, the bacteriological examination should next be undertaken, and the chemical examination should be reserved until the last.

The bacteriological examination should first consist in feeding a number of different species of animals—the larger the number the better—for a day or two exclusively upon the food. White mice, house mice, white rats, young dogs, cats, rabbits, or guinea pigs can be used. If the animals sicken and die they are to be subsequently examined for the presence of pathogenic bacteria. It may happen that none of the animals thus fed will be injured by the food, which fact would not exclude, however, the presence of a germ requiring a specially susceptible animal for a subject.

Another set of animals should be injected with a cold extract of the meat made with sterile water. If the animals die, they are to be examined for pathogenic bacteria. A third set of animals should receive similar injections, though of larger portions, of this aqueous extract which has been previously filtered through sterile porcelain. If the animals die from such injections the same as with unfiltered solutions, it is evident that a soluble bacterial chemical poison is present.

The identification of the toxin or real poison produced by the germ is wholly out of the question. The most that can be done satisfactorily is to obtain,\* as above, a germ-free solution of the poison. It is wholly unnecessary to devote any space in this connection to the detection of the basic bacterial products, the ptomaines, since these bodies are mere cleavage products produced by some and not by other bacteria. Moreover, they are usually but very feebly poisonous, and for that reason they do not hold the prominent position formerly ascribed to them<sup>a</sup>.

A bacteriological examination proper should be made of the original poisonous meat and of all the animals that died either from eating the meat or from the injections of the aqueous extracts. The organism present in the animal, if any, must be isolable directly from the meat. If it happens, as it sometimes has, that the dead animals contain no germs, it is proof that they were killed by a toxin elaborated by a germ in the meat previous to the injection. Cultures from the meat will then reveal the germ, and the effects of its pure cultures should correspond to those observed with the poisonous meat.

To prepare the cultures from the original food, the latter should be cut out with a sterile knife and material should be taken from the inside, thus avoiding all chances of contamination. Several sets of beef-tea tubes and agar plates should be made. One set should be set aside in a Novy anaerobic jar at room temperature; a second similar set should be placed at a temperature of 37° C. A third set should be grown in the presence of air at room temperature, and a like set at a temperature of 37°.

The full details of bacteriological methods must obviously be omitted in this connection. Such work requires a special laboratory and special drill. Those who may be further interested are referred to the works of Abbott, Novy, and Sternberg.

### 3.—PREPARATION OF SAMPLE FOR CHEMICAL EXAMINATION.

In the case of fresh meat, separate the sample as completely as possible from the bones and pass it rapidly and repeatedly through a sausage mill until thorough mixture and complete maceration are obtained. The sample must be kept on ice to prevent decomposition, and all of the determinations should be begun as soon as practicable after the sample is prepared. In the case of canned meats, pass the

\*For detailed methods see Vaughan and Novy, "Cellular Toxins," 4th edition, 1902.

entire contents of a can through a sausage mill as directed above. Remove sausage from the casings and mix by repeated grinding in a sausage mill. Dry that portion of the sample which is not needed for analysis, extract with gasoline which boils below 60° C, allow the gasoline to evaporate spontaneously, and expel the last traces by heating for a short time on the steam bath. Neither the meat nor the separated fat should be heated longer than necessary, owing to the tendency of the latter to decompose. Reserve the fat for examination according to the methods given under the examination of edible fats and oils (page 20). Fat must be kept in a cool place, and its examination finished before it becomes rancid.

#### 4.—DETERMINATION OF WATER. <sup>a</sup>

Dry to constant weight about 2 grams of the macerated sample, in a tared, flat-bottomed dish at the temperature of boiling water. The dish may be of aluminum or platinum, or a tin bottle cap answers admirably for this purpose. On account of the oxidation of the fat, meats may be dried to advantage in a current of hydrogen or *in vacuo*, although satisfactory results are obtained in the above way. Drying usually requires about five hours.

#### 5.—DETERMINATION OF ASH. <sup>a</sup>

Ignite the residue from the determination of water to low redness as long as smoke or inflammable gases are given off. Exhaust the charred mass with 5 or 10 cc of water, transfer to a filter, and wash with hot water till the greater part of the soluble salts are removed. Transfer filter paper and contents to the original dish and ignite at bright red heat till combustion is complete (a white ash can rarely be obtained). Transfer the soluble portion to the dish, add a few drops of ammonium-carbonate solution, evaporate to dryness, heat for a moment in a free flame to very low redness, cool in a desiccator and weigh. Satisfactory results may often be obtained without exhaustion by igniting 0.5 gram of the substance in a porcelain crucible cover.

#### 6.—DETERMINATION OF ETHER EXTRACT.

It has recently been shown that fat can not be completely extracted from meat by means of ether. A complete extraction can be obtained only after digesting the proteids and muscular tissue with pepsin and extracting again with an organic solvent. Voit <sup>b</sup> extracts first with alcohol, to remove the last traces of water, and then with ether in a continuous extractor. This process leaves very little fat in the sample. Comparative results which are satisfactory in all ordinary examinations of meat may be obtained by extracting 2 grams of the dried, finely divided sample with ether for 16 hours in a continuous extractor. Fat may be determined by extracting the ether extract with low boiling-point petroleum ether.

#### 7.—DETERMINATION OF NITROGENOUS SUBSTANCES.

##### (a) TOTAL NITROGEN.

Employ either the Kjeldahl or the Gunning method, using about 2 grams of the sample. The digestion with sulphuric acid should be continued at least 4 hours.

##### (b) COAGULATED PROTEIDS.

Thoroughly exhaust 2 grams of the sample with cold water after extraction with ether, filter, and determine nitrogen in the insoluble residue as directed under "Total nitrogen." Multiply the percentage of nitrogen so obtained by 6.25 for the percent-

<sup>a</sup> See Appendix, p. 149.

<sup>b</sup> Ztschr. f. Biol., 1897, 35, 555.

age of meat fiber or coagulated proteids. (In case the connective tissue is determined, a corresponding correction must be made in the percentage of coagulated proteids.)

(c) DETERMINATION OF CONNECTIVE TISSUE.

Extract 10 grams of the sample with cold water as directed above, then boil the exhausted residue repeatedly with about 100 cc of water until the total extract amounts to about 1 liter. Filter the extract, concentrate by evaporation, and determine the nitrogen content. Multiply the nitrogen so obtained by 5.55 for the percentage of nitrogenous substances of connective tissue.

(d) DETERMINATION OF COAGULABLE PROTEIDS (FOR UNCOOKED MEAT ONLY).

Almost neutralize the filtrate from the coagulated proteids, leaving it still faintly acid, boil until the globulins are coagulated, filter, wash, transfer the filter paper and contents to a Kjeldahl flask, and determine nitrogen as directed above under "Total nitrogen." Multiply the percentage of nitrogen obtained by 6.25 for the percentage of coagulable proteids.

(e) DETERMINATION OF PROTEOSES, PEPTONES, AND GELATIN.

(1) *First method.*

This is a combination of Bömer's<sup>a</sup> method with that of Allen and Searle,<sup>b</sup> as modified by Wiley.<sup>c</sup>

Evaporate the filtrate from the globulins to small volume, add 2 or 3 drops of 1-3 sulphuric acid, and saturate with powdered zinc sulphate. The excess of zinc sulphate added should not be large, as otherwise serious "bumping" is likely to ensue. About 80 grams of the salt are required for each 50 cc of liquid. Allow the coagulated proteids to subside, filter, and wash with a saturated solution of zinc sulphate.

Acidulate the filtrate from the zinc sulphate precipitate with 2 or 3 drops of strong hydrochloric acid, dilute with an equal volume of water, add about 2 cc of liquid bromin, and shake the contents of the flask vigorously. (This can be most conveniently done in a Kjeldahl flask.) If the bromin be all taken up, add more until about 0.5 cc of liquid bromin is left undissolved and the supernatant liquid thoroughly saturated. Allow the mixture to stand over night, decant the supernatant liquid through a filter paper, and wash with water, so directing the jet that the globule of bromin is stirred up and saturates the wash water. Return the filter paper and precipitate to the flask, add the zinc sulphate precipitate and filter paper containing it, and determine the nitrogen as directed under "Total nitrogen." The percentage of nitrogen so found, multiplied by 6.25, gives the percentage of proteoses, peptones, and gelatin, including gelatin peptone.

(2) *Second method.*<sup>d</sup>

Heat the filtrate from albumen and globulins, add a slight excess of tannic acid and a few drops of a saturated solution of alum, allow to cool, filter, and wash with cold water. Heat the filtrate from the tannic-acid precipitate almost to boiling, add an excess of phospho-tungstic acid,<sup>e</sup> separate the precipitated proteids by filtration

<sup>a</sup> Ztschr. anal. Chem., 1895, 5, 562.

<sup>b</sup> The Analyst, 1897, 22, 258-263.

<sup>c</sup> U. S. Dept. Agr., Div. of Chem., Bul. 54.

<sup>d</sup> Mallet, U. S. Dept. of Agr., Div. of Chem., Bul. 54.

<sup>e</sup> Mallet employs two solutions, one containing 50 and the other 100 grams of crystalline phospho-duodeci-tungstic acid dissolved in 1 liter of 2½ percent of hydrochloric acid. He also recommends the addition of sand or pulverized glass to prevent the formation of the coagulated proteids in a dense clot. Owing to the liability of "bumping" in the presence of such substances, however, during the determination of nitrogen it would seem that such addition should be avoided if possible.

and wash with hot water, being careful that the temperature of the solution and wash water shall not be less than 90° C. at any time.

Transfer the filter papers containing the tannic acid and phospho-tungstic acid precipitates to a Kjeldahl flask and determine nitrogen. The nitrogen so obtained multiplied by 6.25 gives the percentage of proteoses, peptones, and gelatin.

#### (f) DETERMINATION OF MEAT BASES.

Deduct from the total nitrogen the sum of the nitrogen obtained in the determination of coagulated proteids, connective tissue, globulin, and proteoses, peptones, and gelatin for the nitrogen of meat bases. Multiply the result by 3.12 for the percentage of meat bases.<sup>a</sup>

### 8.—DETERMINATION OF STARCH (FOR CHOPPED MEAT, SAUSAGE, DEVILED MEAT, ETC.).

#### (a) QUALITATIVE DETERMINATION.

Treat 5 or 6 grams of the sample with boiling water for two or three minutes; cool the mixture, and test the supernatant liquid with iodine solution. In using this test it must be remembered that a small amount of starch may be present as the result of the use of spices. If a marked reaction is given, however, it may be concluded that starch or flour has been added, and a quantitative determination may be made. The above qualitative method may be replaced by a microscopic examination, by which not only the presence of added starch, but also the variety employed, may be determined.

#### (b) QUANTITATIVE DETERMINATION.

The official methods of the association for the determination of starch will not answer for the examination of meat because of the presence in meat of bodies which hold a portion of the cuprous oxid in solution, and thus give results which are too low. In this laboratory Munson examined a series of sausages which contained no starchy material except the spices employed in their manufacture. He obtained less reduced copper from the sausages than from the blank determination of reducing bodies in the malt infusion employed.

#### (1) *Ambühl's method.*<sup>b</sup>

This method has been adopted by Swiss authorities. It is short and convenient, although the results obtained by it are only roughly approximate.

Thoroughly macerate 2 grams<sup>c</sup> of the meat under examination with fifty times its weight of water. Boil for thirty minutes and dilute to 100 cc for every gram of meat employed. Cool an aliquot portion of the liquid, treat with iodine, and compare the depth of color with solutions containing a known amount of the same kind of starch (the variety of starch in the sample is determined microscopically), and boiled for the same length of time.

#### (2) *Mayrhofer's method,*<sup>d</sup> *modified by Bigelow.*<sup>e</sup>

Treat from 10 to 20 grams of the sample under examination (depending upon the amount of starch indicated by the iodine reaction) in a porcelain dish or casserole with 50 cc of an 8 per cent solution of potassium hydroxid and heat the mixture on the water bath until the meat is entirely dissolved. The operation may be hastened by macerating the larger pieces with a glass rod. Add an equal volume of 95 per

<sup>a</sup> See appendix, p. 149.

<sup>b</sup> Pharm. Centralh., 1881, 22, 438; Abstract Ztschr. anal. Chem., 1882, 21, 436.

<sup>c</sup> Ambühl directs that from 2 to 10 grams be employed, according to the size of the meat particles. If the sample be macerated, however, as directed under the preparation of sample, it is unnecessary to employ a large amount.

<sup>d</sup> Forsch. ii. Lebensm., 1896, 3, 141, and 1897, 4, 47.

<sup>e</sup> U. S. Dept. of Agr., Bureau of Chem. Bul. 13, part 10.

cent (by volume) alcohol, mix thoroughly, filter the mixture through an asbestos filter and wash twice with a hot 4 per cent solution of potassium hydroxid in 50 per cent alcohol. Then wash with 50 per cent alcohol until a small portion of the filtrate does not become turbid upon the addition of acid. Return the precipitate and filter to the original vessel and dissolve the precipitate, with the aid of heat, in 60 cc of a normal solution of potassium hydroxid. In the case of sausage with a high starch content a somewhat larger volume of alkali may be required. Acidify the filtrate strongly with acetic acid, dilute to a definite volume, thoroughly mix by shaking, filter through a fluted filter, and precipitate the starch from an aliquot part of the filtrate by means of an equal volume of 95 per cent alcohol (sp. gr. 0.81). Transfer the precipitate to a filter, thoroughly wash with 50 per cent alcohol (by volume), with absolute alcohol, and finally with ether, dry to a constant weight at the temperature of boiling water, and weigh.

#### 9.—DETERMINATION OF GLYCOGEN.

The determination of glycogen has been suggested<sup>a</sup> as a means to detect the presence of horse meat. Recent results indicate that this determination is of limited value because of the fact that glycogen begins to disappear soon after the death of the animal and may entirely disappear after a short lapse of time. No definite conclusions can therefore be derived from the results of this determination, but it is of value as confirmatory.

##### (a) QUALITATIVE METHOD.<sup>b</sup>

Boil 50 grams of the macerated sample with 50 cc of water for from fifteen to thirty minutes. Filter the broth through a moistened filter paper or piece of fine linen. To a portion of the filtrate in a test tube add a few drops of a reagent composed of 2 parts iodine, 4 parts potassium iodide, and 100 parts water. In the presence of a large percentage of horse meat the glycogen contained produces a dark brown color, which is destroyed by heating and reappears on cooling. When starch is present it may be precipitated by two volumes of glacial acetic acid, separated by filtration, and the test for glycogen repeated in the filtrate.

##### (b) QUANTITATIVE METHOD.

The methods for the quantitative examination of glycogen are all tedious to the last degree. Fairly satisfactory results may be obtained by the methods of Brücke,<sup>c</sup> R. Külz,<sup>d</sup> Pflüger,<sup>e</sup> Haywood,<sup>f</sup> and Pflüger and Nerking.<sup>g</sup> Of these the last has been selected as combining a sufficient degree of accuracy with the greatest simplicity and convenience.

Digest 50 grams of finely macerated meat on the water bath with 200 cc of 2 per cent potassium hydroxid until solution is practically complete. Cool the solution, dilute with water to exactly 200 cc, shake and filter. Treat 100 cc of the filtrate with 10 grams of potassium iodide and 1 gram of potassium hydroxid and stir until solution is complete. Add 50 cc of 96 per cent alcohol, and allow to stand until the following day. Then separate the precipitated glycogen by filtration, and wash with a solution containing 1 cc of 73 per cent potassium hydroxid, 10 grams of potassium iodide, 100 cc of water, and 50 cc of 96 per cent alcohol (sp. gr. .81). Wash the glycogen with a mixture of two volumes of 96 per cent alcohol and one volume of water containing about 7 mg of sodium chlorid per liter, dissolve in water, and remove the remaining traces of proteids by the addition of double iodide of mercury and potassium. It is often

<sup>a</sup> Niebel. *Ztschr. ang. Chem.*, 1895, 620.

<sup>b</sup> Courlay and Coremons. *Ztschr. Nahr. Hyg. Waar.*, 1896, 10, 173-174.

<sup>c</sup> *Sitzungsber Acad. Wissensch., Wien*, Bd. 63, II abth., 1871, p. 214.

<sup>d</sup> *Ztschr. f. Biol.*, 4, 169.

<sup>e</sup> *Arch. ges. Physiol.*, 1899, 75, 120-247.

<sup>f</sup> *Jour. Am. Chem. Soc.*, 1900, 22, 85.

<sup>g</sup> *Arch. Physiol.*, 1899, 76, 531-542.

found that the proteids are so completely removed that no precipitate is formed with the double iodid. In such case filtration is not necessary.

Add about 2 mg of sodium chlorid per 100 cc of water, precipitate the glycogen again by means of two volumes of 96 per cent (sp. gr. 0.81) alcohol, filter, wash with 96 per cent alcohol, containing about 7 mg of sodium chlorid per liter, then with absolute alcohol, finally with ether, dry to constant weight, and weigh.

As a control, invert the precipitated glycogen by boiling for three hours with hydrochloric acid diluted with 10 parts of water and determine the reducing sugar by Allihn's method, multiplying the result by 0.9 for percentage of glycogen.

#### 10.—DETERMINATION OF REDUCING SUGAR.

Boil 100 grams of the finely divided meat for fifteen or twenty minutes in a 500-cc graduated flask with a convenient volume of water. Add a few cubic centimeters of normal lead acetate, cool to room temperature, make up to mark with water, and filter through a fluted filter. Evaporate to a small volume as large an aliquot portion of the filtrate as possible, add a saturated solution of sodium sulphate, make up to a definite volume, and filter through a fluted filter. Determine sugar in an aliquot portion of the filtrate by the Allihn method (p. 49).

#### 11.—DETERMINATION OF POTASSIUM NITRATE.

##### (a) METHOD OF SCHLÖSING-WAGNER.<sup>a</sup>

A flask of about 250-cc capacity is provided with a rubber stopper with two holes. Through one of them is passed the stem of a funnel carrying a glass stopcock. The other carries a delivery tube leading to the receiving vessel. The end of the delivery tube is bent so as to pass easily under the mouth of the measuring burette and is covered with a piece of rubber tubing.

Fifty cubic centimeters of saturated ferrous chlorid solution and the same quantity of 10 per cent hydrochloric acid are placed in a flask. The ferrous chlorid solution is prepared by dissolving nails or other small pieces of iron in hot hydrochloric acid and is kept in glass-stoppered flasks of about 50-cc capacity, entirely filled. The contents of one flask is enough for about twelve determinations, and by using the whole content of a flask as soon as possible after opening, all danger of oxidation which would take place in a large flask frequently opened is avoided.

The contents of the flask are boiled until all the air is driven off. The delivery tube is then placed under the measuring tube, which is filled with 40 per cent potassium hydroxid, then a few drops of water are added and the tube is covered with a piece of filter paper. By a careful and quick inversion, the measuring tube can be brought into the vessel receiving it without any danger of air entering.

One hundred grams of the finely macerated meat are extracted by boiling repeatedly with successive small volumes of water, the aqueous solution is concentrated to a small volume, transferred to the funnel, and, with continued boiling, allowed to pass, drop by drop, into the flask. When almost all has run out, the funnel is washed with three 10-cc portions of 10 per cent hydrochloric acid and these portions are allowed to pass, drop by drop, into the flask; the temperature of the surrounding water will soon be imparted to the contents of the tube, and the volume of nitric oxid is read with the tube in such a position that the level of the water within and without the tube coincide.

The amount of nitric oxid present and the corresponding percentage of nitrate may be calculated in the usual way for the given temperature and barometric pressure, or, to avoid computation, the amount of nitrate may be determined by comparison of the

<sup>a</sup>Agr. Chem. Vers. Stat. Halle, p. 50; Wiley, Principles and Practice of Agricultural Analysis, vol. 2, p. 228.

volume of nitric oxid with that evolved by a definite volume (5 to 10 cc) of normal sodium nitrate solution.

(b) PHENOL-SULPHONIC ACID METHOD.<sup>a</sup>

Weigh 1 gram of the sample into a 100-cc flask, add from 20 to 30 cc of water, and heat on the water bath for fifteen or twenty minutes, shaking occasionally. Add 3 cc of a saturated solution of silver sulphate for each per cent of sodium chlorid present, then add 10 cc of lead subacetate and 5 cc of alumina cream, shaking after each addition. Make up to mark with water, and filter through a fluted filter, returning the filtrate to the filter until it runs clear. Evaporate to dryness 25 cc of the filtrate, add 1 cc of phenol-sulphonic acid,<sup>b</sup> mix thoroughly with a glass rod, add 1 cc of water and 3 or 4 drops of concentrated sulphuric acid and heat on a steam bath for two or three minutes, being careful not to raise the temperature sufficiently to char the material. Now add about 25 cc of water and an excess of ammonium hydroxid. Transfer to a 100-cc flask, add 1 or 2 cc alumina cream if not perfectly clear, dilute to mark with water, and filter if necessary.

Prepare a number of 50-cc Nessler tubes, preferably the long, narrow tubes, placing in the first 1 cc of the standard nitrate solution, in the second 2 cc, and so on to 10 cc, then 12 cc, 15 cc, 18 cc, and 20 cc. The comparison of the solution under examination with these tubes will show directly if it comes within this range, in which case it can be read by direct comparison with the various tubes till the one of the exact shade is found. If the color of the solution be darker than any of the tubes prepared as above, it is preferable to dilute as many times as may be necessary to bring the color within this range by transferring 25 cc of the solution to another tube with a pipette and filling up to the mark with distilled water. In this case the reading of the diluted solution in cubic centimeters of standard solution should be multiplied by the number of times the solution under comparison has been diluted. More exact comparisons can be made looking sidewise through the tubes toward a window covered with white paper and shaded from direct sunlight.

The following table prepared by Mr. Given enables one to determine at a glance the percentage of potassium nitrate in a given sample from the number of cubic centimeters of standard solution employed, if the above directions are followed in detail:

*Per cent potassium nitrate.*

Stand- ard so- lution.	Per cent KNO <sub>3</sub> .	Stand- ard so- lution.	Per cent KNO <sub>3</sub> .	Stand- ard so- lution.	Per cent KNO <sub>3</sub> .
cc		cc		cc	
0.7	0.01	14.7	0.21	28.7	0.41
1.4	.02	15.4	.22	29.4	.42
2.1	.03	16.1	.23	30.1	.43
2.8	.04	16.8	.24	30.8	.44
3.5	.05	17.5	.25	31.5	.45
4.2	.06	18.2	.26	32.2	.46
4.9	.07	18.9	.27	32.9	.47
5.6	.08	19.6	.28	33.6	.48
6.3	.09	20.3	.29	34.3	.49
7.0	.10	21.0	.30	35.0	.50
7.7	.11	21.7	.31	35.7	.51
8.4	.12	22.4	.32	36.4	.52
9.1	.13	23.1	.33	37.1	.53
9.8	.14	23.8	.34	37.8	.54
10.5	.15	24.5	.35	38.5	.55
11.2	.16	25.2	.36	39.2	.56
11.9	.17	25.9	.37	39.9	.57
12.6	.18	26.6	.38	40.6	.58
13.3	.19	27.3	.39	41.3	.59
14.0	.20	28.0	.40	42.0	.60

<sup>a</sup> This method is a modification of the one ordinarily employed for determining potassium nitrate in water. It was adapted to the examination of meat by Mr. Arthur Given.

<sup>b</sup> Prepared by mixing together 37 cc of concentrated sulphuric acid, 3 cc of distilled water, and 6 grams of phenol.

## 12. DETECTION OF PRESERVATIVES.

The chemical preservatives commonly used with meat products are borax and boric acid and sulphites. Salicylic and benzoic acids are occasionally used, and formaldehyde is said to be used, although the writer has failed to detect its presence in meat preparations. The general methods for the detection of these preservatives are given on pages 107 and 110. A few special methods are described below. In general, preservatives may be separated from meat by digesting a few minutes in warm water, made slightly acid or slightly alkaline according as the nature of the preservative is basic or acid.

## (a) BORAX AND BORIC ACID.

If present in noticeable amounts, boric acid may be detected in meat products by heating 20 grams of the sample a few minutes in about 100 cc water acidified with 6 or 8 cc of concentrated hydrochloric acid and testing with turmeric paper as directed on page 110. If no action is obtained by this method, about 20 grams of the sample should be made alkaline with calcium hydroxid, ignited, and the ash tested as directed under preservatives.

## (b) SULPHUROUS ACID.

The distillation method for the detection of sulphurous acid (see page 107) will answer for the examination of meat, but mere traces should be ignored. According to Ostertag,<sup>a</sup> the microscopic examination of meat that has been preserved with sodium or calcium sulphite often discloses the presence of crystals of sodium or calcium sulphate, due to partial oxidation of the sulphite.

In the absence of chlorids and nitrates Kämmerer<sup>a</sup> employs potassium iodate paper in the following manner: Place the sample of meat on potassium iodate paper and moisten it with dilute sulphuric acid (1:8) free from oxids of nitrogen. In the presence of even minute traces of sulphites a deep-blue color is immediately formed, while in the absence of sulphites only a faint-blue color is produced, and that after a considerable time. This method is of limited application, since it can not be used with meats containing salt or saltpeter.

13. DETECTION OF COLORING MATTER.<sup>b</sup>

Sausages and other preparations in which chopped meat is employed rapidly become discolored on exposure to the air. This change does not take place to a marked extent with meat that has been cured in a pickle containing saltpeter. With fresh chopped meat, and sometimes with corned meat, especially that cured without saltpeter, coloring matter is sometimes added to prevent the change of color which would naturally take place. Aniline dyes and cochineal carmine are ordinarily employed for this purpose, though in some instances vegetable colors have been detected in the form of lakes. The coloring matter may often be extracted by heating for 15 or 20 minutes with 50 per cent alcohol, 50 per cent glycerin slightly acidified, a mixture of alcohol and glycerin,<sup>c</sup> ammonium hydroxid, or a 5 per cent solution of sodium salicylate<sup>d</sup> in water. Approximately equal weights of meat and solvent may be used.

In case the filtered extract by any of these methods is colored red or deep yellow, it should be evaporated nearly to dryness, slightly acidified with hydrochloric acid, and boiled a few minutes after the addition of a thread of fat free wool. If the wool is dyed, it may be examined as directed by the referee on coloring matter. If the wool is not dyed, the solution is examined spectroscopically.

<sup>a</sup> Handbuch der Fleischbeschau, 3 ed., p. 826.

<sup>b</sup> See appendix, p. 149.

<sup>c</sup> Klinger and Bujard, Ztschr. ang. Chem., 1891, 515.

<sup>d</sup> Spaeth, Pharm. Centralh., 1897, 38, 884.



If too dilute, the solution may often be concentrated by precipitating the coloring matter as a lake,<sup>a</sup> allowing it to settle, decanting off the water, dissolving in hydrochloric acid and making alkaline with ammonia.

In extracting with 50 per cent alcohol, the proteids of the meat are coagulated, with the formation of a pale, almost white, color. If the meat is not discolored during this extraction, it is probable that some foreign color is present.<sup>b</sup>

Marpmann<sup>b</sup> examines sausages microscopically for the presence of coloring matter after dehydrating with alcohol and xylol consecutively, removing the xylol with carbon tetrachlorid, and immersing in cedar oil until the natural colors of the meat have disappeared.

## (B) MEAT EXTRACTS.

### 1.—PREPARATION OF SAMPLE.

Liquid and semiliquid meat extracts and similar preparations should be removed from the container and thoroughly mixed before sampling. With many liquid preparations a sediment is found in the bottom of the container which will be overlooked if great care is not taken.

### 2.—DETERMINATION OF WATER.

Follow directions given on page 10, employing about 2 grams of powdered preparations, about 3 grams of preparations of pasty consistency, and from 5 to 10 grams of liquid extracts, according to the solid contents. Dry the powdered preparations directly without admixture. Dissolve the pasty preparations in water and dry with sufficient ignited asbestos or pumice stone to absorb the solution. Tin or lead dishes or Hofmeister glass dishes, are often convenient with samples in which the residue is to be extracted for fat, as the dishes may be cut or broken and placed in the extraction tube with the sample.

### 3.—DETERMINATION OF ASH.

Proceed as directed on page 10. In case of pasty preparations, add sufficient water to effect solution and evaporate to dryness in order that the solids may be distributed evenly over the bottom of the dish.

### 4.—DETERMINATION OF FAT.

Transfer the residue from the determination of water to the tube of a continuous extraction apparatus, wash any fat adhering to the dish into the tube with ether, and extract with ether sixteen hours.

### 5.—DETERMINATION OF NITROGENOUS SUBSTANCES.

#### (a) TOTAL NITROGEN.

Employ either the Kjeldahl or the Gunning method.

#### (b) DETERMINATION OF MEAT FIBER.<sup>c</sup>

Dissolve in cold water 5 grams of powdered preparations, from 8 to 10 grams of extracts of pasty consistency, or from 20 to 25 grams of fluid extracts; filter and wash with cold water. Transfer the filter paper and contents to a Kjeldahl flask and determine nitrogen as directed under total nitrogen. In case of a large amount of insoluble matter, make up to a definite volume, filter through a fluted filter paper,

<sup>a</sup>Bremer, Forschungsber., 1897, 4, 45.

<sup>b</sup>Ztschr. ang. Mikr., 1895, 1, 12.

<sup>c</sup>Allen, Com. Org. Anal., 2d ed., vol. 4, p. 324.

and determine nitrogen in an aliquot portion of the filtrate; then deduct the percentage of nitrogen in the total filtrate from the percentage of total nitrogen for the percentage of nitrogen in meat fiber. Multiply the percentage of nitrogen by 6.25 for the percentage of meat fiber.

(c) DETERMINATION OF COAGULABLE PROTEIDS.

Make the filtrate (as large an aliquot portion as practicable when the nitrogen of meat fiber has been determined by difference) from meat fiber slightly acid (if not already acid), adding acetic acid or sodium hydroxid as may be required, boil for two or three minutes, cool to room temperature, dilute to 500 cc and filter through a fluted filter.<sup>a</sup>

Determine nitrogen in 50 cc of the filtrate by means of the Kjeldahl or Gunning method. Ten times the nitrogen so obtained deducted from the percentage of soluble nitrogen (which in turn is obtained by deducting percentage of nitrogen occurring as meat fiber from the total nitrogen) gives the percentage of nitrogen contained in albumin and globulins. Multiply this figure by 6.25 for the percentage of coagulable proteids in the sample.

(d) DETERMINATION OF SYNTONIN.

Exactly neutralize the filtrate from the determination of coagulable proteids with sodium hydroxid, using litmus as indicator, and allow to stand until the precipitate settles. If only a small amount of syntonin is precipitated, it may be separated with an ordinary filter, washed with water, and its nitrogen content determined by means of the Kjeldahl or Gunning method. If present in any considerable quantity, dilute to a definite volume, filter through a fluted filter, and determine nitrogen in 50 cc of the filtrate.

The nitrogen thus obtained (calculated to total volume) is deducted from the nitrogen in the filtrate from the globulins for the syntonin nitrogen. This multiplied by 6.25 gives syntonin.

(e) DETERMINATION OF PROTEOSES, PEPTONES, AND GELATIN.<sup>b</sup>

If it be desired to group these bodies together, proceed as directed under (e), page 11, unite the two precipitates and make a single determination of nitrogen. The percentage of these bodies can not be determined by using aliquot parts and deducting the nitrogen content of the filtrate from the bromin precipitate from that of the filtrate from the determination of syntonin, because of the decomposing effect exerted by bromin on nitrogen compounds. Experiments in this laboratory also indicate that the aliquot portions of the filtrate from the determination of syntonin can not be used separately for the zinc-sulphate precipitate and the bromin precipitate for the same reason. Although bromin precipitates peptones and zinc sulphate does not, Trescott found, in the examination of a large number of meat extracts when working with aliquot portions of the same solution, that more nitrogen was precipitated by zinc sulphate than by bromin.

(f) DETERMINATION OF PROTEOSES AND GELATIN.<sup>c</sup>

Evaporate the filtrate from the determination of syntonin (as large an aliquot portion of the filtrate as is practicable when the percentage of syntonin is determined

<sup>a</sup>The filtering and washing of coagulated proteids are always tedious and unsatisfactory and sometimes almost impossible. The work is greatly simplified, therefore, by passing through a fluted filter and employing aliquot parts of the filtrate, as by this means the complete filtration and washing of precipitates is made unnecessary.

<sup>b</sup>Allen, *The Analyst*, 1897, **22**, 258; *Com. Org. anal.*, 2d edition, vol. 4, p. 325.

<sup>c</sup>Bömer, *Ztschr. anal. Chem.*, 1895, **5**, 562; also Mallet, *U. S. Dept. Agr., Div. of Chem., Bul.* 54.

by difference, as suggested by the writer) to a small volume and saturate with zinc sulphate. About 85 grams of powdered zinc sulphate are necessary for the saturation of 50 cc of the liquid at ordinary laboratory temperature. The liquid must be fully saturated with the salt, but a large excess should be avoided, as it is likely to cause "bumping" in the subsequent determination of nitrogen in the solution. Let stand several hours, filter, and wash the precipitate with saturated zinc sulphate. In case the precipitate is voluminous, which rarely happens, the mixture may be made up to a definite volume with saturated zinc sulphate, filtered, the nitrogen may be determined in an aliquot portion of the filtrate, and the nitrogen of the precipitated proteids determined by difference.

(g) DETERMINATION OF PEPTONES. <sup>a</sup>

Dilute the filtrate from the zinc-sulphate precipitate of proteoses and gelatin with an equal volume of water, add bromin until a globule of from 0.5 cc to 1 cc remains undissolved after the liquid is saturated, and allow to stand over night. Filter, wash with cold water, directing the jet to the globule of bromin so as to keep the wash water saturated. Transfer the filtered precipitate to a Kjeldahl flask and determine nitrogen.

(h) DETERMINATION OF GELATIN.

*Stutzer's method<sup>b</sup> modified by Bigelow.<sup>c</sup>*

Boil 10 grams of the sample for a few minutes with water; filter, wash, and evaporate the filtrate to dryness in a porcelain dish of about 10 cm diameter, after the addition of about 20 grams of sand which has been freed from dust by sifting, and thoroughly ignited. Exhaust the residue with four 100-cc portions of absolute alcohol, and pass the supernatant liquid through an asbestos filter which rests on a porous plate of about 4 cm diameter, in a funnel. The funnel is surrounded by pounded ice and attached to an aspirator, by means of which gentle and gradually increasing suction may be applied. Take care to transfer as little as possible of the insoluble residue to the filter. Then extract the residue repeatedly with 100-cc portions of a mixture containing 100 cc of 95 per cent alcohol (sp. gr. 0.81), 300 grams of ice, and 600 grams of cold water, taking care that the temperature shall not exceed 5° C. at any time. Continue the extraction until the various portions of solvent used are entirely colorless. Filter the extract through the funnel employed for the alcohol extract. Finally, return the asbestos filter to the beaker which contains the exhausted residue and thoroughly extract the whole with boiling water. Receive the hot-water extract in a Kjeldahl flask, determine nitrogen, and multiply the percentage of nitrogen so obtained by 5.55 for the percentage of gelatin and gelatin peptone.

(i) PROTEOSES.

Deduct the nitrogen in the gelatin precipitate (h) from that of the proteose and gelatin precipitate. This multiplied by 6.25 gives the percentage of proteoses.

(j) MEAT BASES.

Deduct from the total nitrogen (a) the sum of the nitrogen in (b), (c), (d), (f), and (g). Multiply the difference by 3.12 for meat bases.

6.—DETERMINATION OF GLYCOGEN.

Proceed as directed on page 13.

7.—DETECTION OF PRESERVATIVES.

Proceed as directed under Preservatives, page 107.

<sup>a</sup> Allen Com. Org. anal, 2nd Ed., vol. 4, p. 320.

<sup>b</sup> Ztschr. anal. Chem., 1895, 34, 568.

<sup>c</sup> U. S. Dept. of Agr., Bureau of Chem., Bul. 13, Part 10.

## II. EDIBLE OILS AND FATS.

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## 1.—GENERAL DISCUSSION.

In working with oils and fats the same methods of examination largely apply, except in preparation of the sample.

The solid fats should first be melted, thoroughly mixed, and then filtered by means of a hot-water funnel or similar apparatus. Samples for the different determinations are taken from this melted homogeneous mass. The specific gravity must be taken at some temperature above the melting point of the fat. The boiling point of water has been largely used, and, although there are inherent errors in such a method,<sup>a</sup> it probably gives the most satisfactory results for practical work.

In the Maumené test, fats require a higher initial temperature than oils.

With oils in most cases the sample for analysis requires no preliminary treatment, except that in case of impurities the oil should be filtered.

Oil and fat should always be kept in a cool place, otherwise they will soon become rancid, which will affect more or less the physical and chemical constants.<sup>b</sup> The iodine number decreases with rancidity, while specific gravity, index of refraction, and acetyl value increase. Too much confidence must not be placed in negative results obtained in a single determination, but only upon making a complete quantitative as well as qualitative examination can a reliable judgment of purity be made.

Oils and fats being variable mixtures of glycerids of the fatty acids, their physical and chemical constants vary within limits fairly well established from analytical data. However, too much dependence must not be placed upon the more common determinations, as it is easy to make such mixtures of either fats or oils as will satisfy the ordinary requirements as to specific gravity, index of refraction, heat with sulphuric acid, iodine absorption, and saponification value.

The melting point of the fats and the fatty acids is difficult to determine, because they are mixtures of glycerids or acids, substances which have widely varying melting points. A wide difference has resulted from the varied usage of different analysts, and results obtained by exactly the same method are the only ones that are strictly comparable. For fats Wiley's method of determining melting point has been adopted by the Association of Official Agricultural Chemists.

For the free fatty acids obviously this will not do, and the capillary tube was chosen as being a method most generally used and giving the most satisfactory results.

2.—DETERMINATION OF SPECIFIC GRAVITY.<sup>c</sup>

## (a) DETERMINATION AT 15.5° C.

Determine the specific gravity of oils at 15.5° C. by the use of a pycnometer, Westphal balance,<sup>d</sup> or accurately graduated hydrometer.<sup>e</sup>

If determined at room temperature, the following formula may be used to calculate the specific gravity at 15.5° C.:<sup>f</sup>

$$G = G' + .00064 (T - 15.5 \text{ C.}).$$

$$G = \text{sp. gr. at } 15.5^\circ.$$

$$G' = \text{sp. gr. at } T.$$

$$0.00064 = \text{mean correction for } 1^\circ \text{ C.}$$

<sup>a</sup> E. E. Ewell, U. S. Dept. Agr., Div. Chem., Bul. 62, p. 125.

<sup>b</sup> E. Spaeth, Ztschr. anal. Chem., 1896, **35**, 471-493; C. A. Browne, Jour. Am. Chem. Soc., 1899, **21**, 989-994.

<sup>c</sup> See appendix, p. 149.

<sup>d</sup> C. A. Crampton, U. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 4, p. 438.

<sup>e</sup> Accurately made hydrometers reading from sp. gr. 0.900 to 0.940 at 15.5° C. will satisfy every requirement of accuracy and speed.

<sup>f</sup> Allen, Com. Org. Anal., 3d ed., vol. 2, pt. 1, p. 33; Winton, Conn. Expt. Sta. Rept., pt. 2, 1900, p. 149.

This is only approximately correct, as the correction varies for different oils, but will satisfy ordinary requirements. If a higher degree of accuracy is desired, the factors given in the following table may be employed, but to obtain the best results the determination must be made at standard temperature.

*Factors for calculating specific gravity.<sup>a</sup>*

Oil.	Correction for 1° C.	Observer.
Cod-liver oil.....	0.000646	A. H. Allen.
Lard oil.....	.000658	C. M. Wetherill.
Olive oil.....	.000629	C. M. Stillwell.
Arachis oil.....	.000655	A. H. Allen.
Rape oil.....	.000620	Do.
Sesame oil.....	.000624	Do.
Cotton-seed oil.....	.000629	Do.
Cocoonut olein.....	.000665	Do.

The following table gives correction for solid fats:<sup>b</sup>

*Factors for calculating specific gravity.*

Fats.	Correction for 1° C.
Cocoa butter.....	0.000717
Tallow.....	.000675
Lard.....	.000650
Butter fat.....	.000617
Cocoonut stearins.....	.000674
Cocoonut oil.....	.000642
Palmnut oil.....	.000657

(b) DETERMINATION AT THE TEMPERATURE OF BOILING WATER.<sup>c</sup>

(1) *Standardization of flasks.*

*First method.*—Use a small specific gravity flask of from 25 to 30 cc capacity. The flask is to be thoroughly washed with hot water, alcohol, and ether, and then dried. After cooling in a desiccator the weight of the flask and stopper is accurately determined.

The flask is filled with freshly boiled and still hot distilled water and placed in a bath of pure distilled water. The water of the bath is kept in brisk ebullition for thirty minutes, any evaporation from the flask being replaced by the addition of boiling distilled water. The stopper, previously heated to 100°, is then inserted, the flask removed, wiped dry, and after it has nearly cooled to room temperature placed in the balance, and weighed when balance temperature is reached.

*Second method.*<sup>d</sup>—The following formula may be used for calculating the weight of water ( $W^T$ ) which a given flask will hold at  $T^\circ$  (weighed in air with brass weights at the temperature of the room) from the weight of water ( $W^t$ ) (weighed in air with brass weights at the temperature of the room) contained therein at  $t^\circ$ :

$$W^T = W^t \frac{d^T}{d^t} [1 + \gamma (T - t)]$$

$d^T$  = the density of water at  $T^\circ$ .

$d^t$  = the density of water at  $t^\circ$ .

$\gamma$  = the coefficient of cubical expansion of glass.<sup>e</sup>

<sup>a</sup> Allen, Com. Org. Anal., 3d ed., vol. 2, pt. 1, p. 33.

<sup>b</sup> Allen, Com. Org. Anal., 3d ed., vol. 2, pt. 1, p. 32.

<sup>c</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 33.

<sup>d</sup> E. E. Ewell, U. S. Dept. Agr., Div. Chem., Bul. 62, p. 125.

<sup>e</sup> This factor is commonly given as 0.000026, but it varies considerably. Schulze (Ztschr. anal. Chem., 1882, 21, 167-177) found the glass used by him varied from 0.0000288 to 0.0000305; an average of these is 0.0000296. Ewell has used 0.000028 in his work, U. S. Dept. of Agr., Div. of Chem., Bul. 62, p. 121.

(2) *Determination.*

*Weight of fat at the temperature of boiling water.*—The flask is rinsed with alcohol and ether, and dried for a few minutes at the temperature of boiling water. It is filled with the dry, hot, fresh-filtered fat, which should be entirely free from air bubbles, replaced in the water bath, and kept for thirty minutes at the temperature of boiling water. The stopper, previously heated to 100° C, is inserted, the flask removed, wiped dry, placed in the balance after it has nearly cooled to room temperature, and weighed when the balance temperature is reached. The weight of fat having been determined, the specific gravity is obtained by dividing it by the weight of water previously found.

Example:	Grams.
Weight of flask, dry .....	10.0197
Weight of flask, plus water .....	37.3412
Weight of water .....	27.3215
Weight of flask, plus fat .....	34.6111
Weight of fat .....	24.5914
Specific gravity = $24.5914 \div 27.3215 = 0.90008$ .	

The weight of the flask dry and empty may be used constantly if great care be taken in handling and cleaning the apparatus, but the weight of water at boiling temperature must be determined under the barometric conditions prevailing at the time the determination is made.

Example:	Grams.
Weight of flask, dry and empty .....	10.0028
Weight of flask after three weeks' use .....	10.0030

3.—DETERMINATION OF INDEX OF REFRACTION. <sup>a</sup>

Determine the index of refraction with any standard instrument, oils being read at 15.5° C. and fats at 40° C.

The temperature must be controlled with great care, and in accurate work the readings should be taken at standard temperature. The readings of the Zeiss butyrorefractometer can be reduced to standard temperature by following formula:<sup>b</sup>

$$R = R' + .55 (T' - T).$$

in which R is the reading reduced to T, R' the reading at Temp. T, T the standard temperature, and .55 the correction for 1° C. in scale divisions. With oils the factor .58 is substituted in the formula for .55, since they have a higher index of refraction.

To calculate to standard temperature the readings of the instruments which give index of refraction directly the factor 0.000365 may be used. As the temperature rises the refractive index falls. Example: The refractive index of a butter fat determined at 32.4° C. = 1.4540 is reduced to 25° C., as follows:  $32.4 - 25 = 7.4$ ;  $0.000365 \times 7.4 = 0.0027$ ; it is then  $1.4540 + 0.0027 = 1.4567$ .

The instrument used should be set with distilled water at 18°C., the theoretical refractive index of water at that temperature being 1.3330. In the determination above given the refractive index of pure water measured 1.3300; hence the above numbers should be corrected for theory by the addition of 0.0030, making the corrected index of the butter fat mentioned at the temperature given 1.4597.

The index of refraction varies greatly with the specific gravity, increasing as it increases. In abnormal results it is often well to see if the specific refractive power<sup>c</sup>

<sup>a</sup> See appendix, p. 150.

<sup>b</sup> Wiley, Prin. and Prac. of Agri. Anal., vol. 3, p. 341. Winton, Conn. Expt. Sta. Rept., 1900, pt. 2, p. 142.

<sup>c</sup> Landolt, Ber., 1882, 15, 1031. C. A. Browne, Jour. Am. Chem. Soc., 1899, 21, 991.

is different from the normal. Calculate the specific refractive power from the formula  $\frac{N-1}{D}$ <sup>a</sup>, in which N equals the refractive index and D the specific gravity. Always state temperature at which the determinations were made.

(a) ABBE'S REFRACTOMETER.

A later and much improved model of the Abbe instrument, in which arrangements are made for controlling the temperature, the weakness of the older form,<sup>b</sup> is described in Benedikt.<sup>c</sup>

(b) ZEISS BUTYRO-REFRACTOMETER.<sup>d</sup>

Place the instrument (fig. 1) upon a table where diffuse daylight or any form of artificial light can be readily admitted for illumination. Supply through nozzle D a

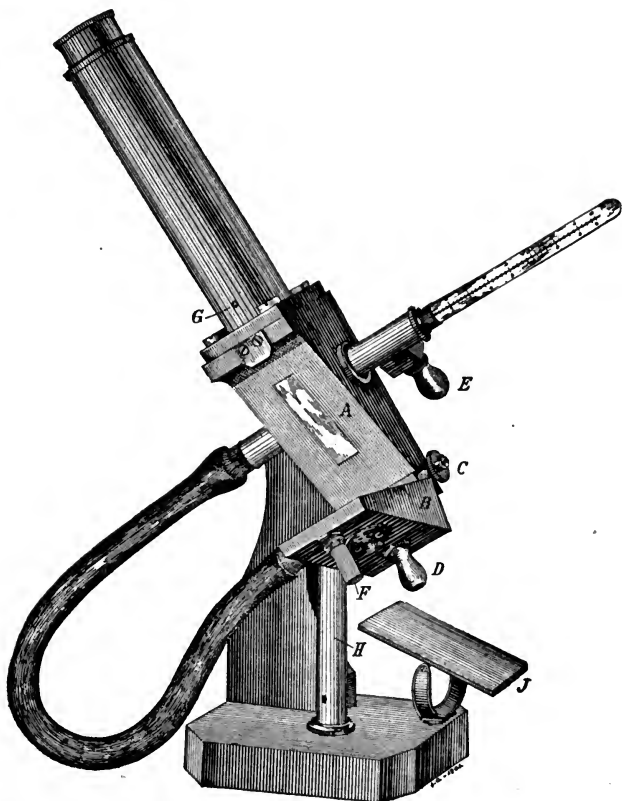


FIG. 1.—Zeiss's butyro-refractometer.

stream of water of constant temperature. Then open the prism casing by giving to the pin F a half turn. The surfaces of the prism must now be cleaned with the greatest care, which is best done by applying soft linen moistened with ether. Now

<sup>a</sup>H. R. Procter, Jour. Soc. Chem., Ind., 1898, **17**, 1021-1026, has shown that the Lorenz formula  $\frac{N^2-1}{(N^2+2)D}$  gives much more satisfactory results than  $\frac{N-1}{D}$ , and gives table for calculation.

<sup>b</sup>For a description of the older form of the Abbe instrument, see U. S. Dept. Agr., Div. Chem., Bul. 46, revised, p. 49.

<sup>c</sup>Anal. der. Fette u. Wach., 3d ed., p. 105.

<sup>d</sup>Wiley, Prin. and Prac. Agr. Anal., vol. 3, pp. 339-341. Also description by manufacturer.

melt the sample of fat and pour the clear fat through a filter, allowing the first two or three drops to fall on the surface of the prism contained in casing B (oils must be filtered if turbid). For this purpose the apparatus should be raised with the left hand, so as to place the prism surface in a horizontal position. Then press B against A and bring F back into its original position by turning it in the opposite direction. Adjust the mirror until it gives the sharpest reading. If the reading be not distinct after running water of a constant temperature through the instrument for some time, the fat is not evenly distributed on the surfaces of the prism and the process must be repeated. The instrument should be carefully adjusted by means of the standard fluid which is supplied. As the index of refraction is greatly affected by temperature, care must be used to keep it constant.

The following table can be used to convert the degrees of the instrument into refractive indices:

*Butyro-refractometer readings and indices of refraction.*<sup>a</sup>

Reading.	Index of refraction.	Reading.	Index of refraction.	Reading.	Index of refraction.	Reading.	Index of refraction.
40.0	1,4524	50.0	1,4593	60.0	1,4659	70.0	1,4723
40.5	1,4527	50.5	1,4596	60.5	1,4662	70.5	1,4726
41.0	1,4531	51.0	1,4600	61.0	1,4665	71.0	1,4729
41.5	1,4534	51.5	1,4603	61.5	1,4668	71.5	1,4732
42.0	1,4538	52.0	1,4607	62.0	1,4672	72.0	1,4735
42.5	1,4541	52.5	1,4610	62.5	1,4675	72.5	1,4738
43.0	1,4545	53.0	1,4613	63.0	1,4678	73.0	1,4741
43.5	1,4548	53.5	1,4616	63.5	1,4681	73.5	1,4744
44.0	1,4552	54.0	1,4619	64.0	1,4685	74.0	1,4747
44.5	1,4555	54.5	1,4623	64.5	1,4688	74.5	1,4750
45.0	1,4558	55.0	1,4626	65.0	1,4691	75.0	1,4753
45.5	1,4562	55.5	1,4629	65.5	1,4694	75.5	1,4756
46.0	1,4565	56.0	1,4633	66.0	1,4697	76.0	1,4759
46.5	1,4569	56.5	1,4636	66.5	1,4700	76.5	1,4762
47.0	1,4572	57.0	1,4639	67.0	1,4704	77.0	1,4765
47.5	1,4576	57.5	1,4642	67.5	1,4707	77.5	1,4468
48.0	1,4579	58.0	1,4646	68.0	1,4710	78.0	1,4771
48.5	1,4583	58.5	1,4649	68.5	1,4713	78.5	1,4774
49.0	1,4586	59.0	1,4652	69.0	1,4717	79.0	1,4777
49.5	1,4590	59.5	1,4656	69.5	1,4720	79.5	1,4780

<sup>a</sup> Winton, Conn. Expt. Sta., Rept., 1900, pt. 2, p. 143.

#### 4.—DETERMINATION OF IODIN ABSORPTION, HÜBL'S METHOD.<sup>a</sup>

##### (a) PREPARATION OF REAGENTS.

*Iodin solution.*—Dissolve 25 grams of pure iodine in 500 cc of 95 per cent alcohol. Dissolve 30 grams of mercuric chlorid in 500 cc of 95 per cent alcohol. The latter solution, if necessary, is filtered, and then the two solutions are mixed. The mixed solution should be allowed to stand twelve hours before using.

*Decinormal sodium thiosulfate solution.*—Dissolve 24.8 grams of chemically pure sodium thiosulfate freshly pulverized as finely as possible and dried between filter or blotting paper, and dilute with water to 1 liter at the temperature at which the titrations are to be made.

*Starch paste.*—One gram of starch is boiled in 200 cc of distilled water for ten minutes and cooled to room temperature.

*Solution of potassium iodid.*—One hundred and fifty grams of potassium iodid are dissolved in water and made up to 1 liter.

*Solution of potassium bichromate.*—Dissolve 3.874 grams of chemically pure potassium bichromate in distilled water and make the volume up to 1 liter at the temperature at which the titrations are to be made. The bichromate solution should be checked against pure iron.

<sup>a</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 50.



## (b) DETERMINATION.

(1) *Standardizing the sodium thiosulfate solution.*

Place 20 cc of the potassium bichromate solution, to which has been added 10 cc of the solution of potassium iodid, in a glass-stoppered flask. Add to this 5 cc of strong hydrochloric acid. Allow the solution of sodium thiosulfate to flow slowly into the flask until the yellow color of the liquid has almost disappeared. Add a few drops of the starch paste, and with constant shaking continue to add the sodium thiosulfate solution until the blue color just disappears. The number of cubic centimeters of thiosulfate solution used multiplied by 5 is equivalent to 1 gram of iodine.

Example: Twenty cubic centimeters of bichromate solution required 16.2 cc sodium thiosulfate; then  $16.2 \times 5 = 81 =$  number cubic centimeters of thiosulfate solution equivalent to 1 gram of iodine. Then 1 cc thiosulfate solution  $= 0.0127$  gram of iodine. Theory for decinormal solution of sodium thiosulfate 1 cc  $= 0.0127$  gram of iodine.

(2) *Weighing the sample.*<sup>a</sup>

Weigh about 1 gram of fat or 0.500 gram of oil<sup>b</sup> on a small watch crystal<sup>c</sup> or by other suitable means. The fat is first melted, mixed thoroughly, poured onto the crystal and allowed to cool.

Introduce the watch crystal into a wide-mouth 16-ounce bottle with ground-glass stopper.

(3) *Absorption of iodine.*

The fat or oil in the bottle is dissolved in 10 cc of chloroform. After complete solution has taken place, 30 cc of the iodine solution are added in the case of fats, or from 40 to 50 cc<sup>d</sup> in the case of oils. Place the bottle in a dark place and allow to stand, with occasional shaking, for three hours.<sup>e</sup> This time must be closely adhered to in order to get good results. The excess of iodine should be at least as much as is absorbed.

(4) *Titration of the unabsorbed iodine.*

Add 20 cc of the potassium iodid solution, and then 100 cc of distilled water to the contents of the bottle. Wash any iodine which may be noticed upon the stopper back into the bottle with the potassium iodid solution. Titrate the excess of iodine with the sodium thiosulfate solution, which is added gradually, with constant shaking, until the yellow color of the solution has almost disappeared. Add a few drops of starch paste, and continue the titration until the blue color has entirely disappeared. Toward the end of the reaction stopper the bottle and shake violently, so that any iodine remaining in solution in the chloroform may be taken up by the potassium iodid solution. The excess of sodium thiosulfate solution should be sufficient to prevent a reappearance of any blue color in the flask for five minutes.

(5) *Setting the value of iodine solution by thiosulfate solution.*

At the time of adding the iodine solution to the fat, two bottles of the same size as those used for the determination should be employed for conducting the operation described above, but without the presence of any fat. In every other respect the

<sup>a</sup>The writer has found it unsatisfactory to weigh so small amounts of fat in flask as directed in the A. O. A. C. methods.

<sup>b</sup>With drying oils which have a very high absorbent power, 0.100 to 0.200 gram should be taken.

<sup>c</sup>See appendix, p. 150.

<sup>d</sup>F. Ulzer, Jour. Soc. Chem. Ind., 1898, 17, 276, says iodine should be in excess about twice the amount that is absorbed. The solution loses strength with age, but can be used so long as 35 cc of decinormal thiosulfate neutralize 25 cc iodine solution.

<sup>e</sup>The time allowed does not give the complete iodine absorption power of an oil or fat and can not be compared with determinations where six to twelve hours have been used. It gives very satisfactory comparative results, but the time factor must be very closely adhered to.

performance of the blank experiments should be just as described. These blank experiments must be made each time the iodine solution is used.

Example blank determinations: Forty cc iodine solution required 62.05 cc of sodium thiosulphate solution. Forty cc iodine solution required 62.15 cc of sodium thiosulphate solution. Mean, 62.1 cc.

Per cent of iodine absorbed:

Weight of fat taken.....	grams..	1.0479
Quantity of iodine solution used .....	cubic centimeters..	40.0
Thiosulfate equivalent to iodine used .....	do....	62.1
Thiosulfate equivalent to remaining iodine.....	do....	30.2
Thiosulfate equivalent to iodine absorbed.....	do....	31.9
Per cent of iodine absorbed, $31.9 \times 0.0124 \times 100 \div 1.0479 = 37.75$ .		

#### 5.—DETERMINATION OF SAPONIFICATION NUMBER AND SOLUBLE AND INSOLUBLE ACIDS.<sup>a</sup>

The saponification number, and soluble and insoluble acids, are determined in one sample by the following method:

##### (a) PREPARATION OF REAGENTS.<sup>b</sup>

*Standard sodium hydroxide solution.*—A decinormal solution of sodium hydroxide is used. Each cubic centimeter contains 0.0040 gram of sodium hydroxide and neutralizes 0.0088 gram of butyric acid ( $C_4H_8O_2$ ).

*Alcoholic potash solution.*—Dissolve 40 grams of good potassium hydroxide in 1 liter of 95 per cent redistilled alcohol.<sup>c</sup> The solution must be clear and the potassium hydroxide free from carbonates.

*Standard acid solution.*—Prepare accurately a half normal solution of hydrochloric acid.

*Indicator.*—Dissolve 1 gram of phenolphthalein in 100 cc of 95 per cent alcohol.

##### (b) WEIGHING OF SAMPLE.

The saponification is carried on in a wide-mouth Erlenmeyer flask holding from 250 to 300 cc. These are cleaned by thoroughly washing with water, alcohol, and ether, wiped perfectly dry on the outside, and heated for one hour at the temperature of boiling water. The flasks are then placed on a tray, covered with a silk handkerchief, and allowed to cool. They must not be wiped with a silk handkerchief within fifteen or twenty minutes of the time they are weighed.

About 5 grams of the melted fat, which has been filtered, is run in by means of a pipette, and after cooling the flask and contents are again weighed.<sup>d</sup>

##### (c) KOETSTORFER OR SAPONIFICATION NUMBER.<sup>e</sup>

Measure 50 cc of the alcoholic potash solution into the flask by means of a burette or pipette, which is allowed to drain a definite time. Connect the flask with a reflux<sup>f</sup> condenser and boil for thirty minutes, when the fat is completely saponified. Cool the flask and titrate with half-normal hydrochloric acid, using phenolphthalein as indicator. The Koetstorfer number (milligrams of potassium hydroxide required to saponify 1 gram of fat) is obtained by subtracting the number of cubic centimeters of hydrochloric acid used to neutralize the excess of alkali after saponification

<sup>a</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 47.

<sup>b</sup> See appendix, p. 150.

<sup>c</sup> The alcohol should be redistilled from potassium hydroxide on which it has been standing for some time, or with which it has been boiled for some time, using a reflux condenser.

<sup>d</sup> See appendix, p. 150.

<sup>e</sup> Chiefly of value in oil work in the detection of rape-oil, resin, and paraffin products.

<sup>f</sup> Almost any sort of a reflux condenser will do. A small funnel placed in the mouth of the flask is perfectly satisfactory and very convenient.

from number of cubic centimeters necessary to neutralize the 50 cc of alkali added, multiplying the result by 28.06 (mg. potassium hydroxid per cubic centimeter) and dividing by the number of grams of fat used.

To calculate the saponification equivalent<sup>a</sup> divide 56,100 by the saponification number, the saponification equivalent being the number of grams of fat saponified by one equivalent of potassium hydroxid, or 56.1 grams. There is no advantage in stating it in this way, and for sake of uniformity, the Koetstorfer number being more generally used, it would seem advisable to adopt it.

(d) SOLUBLE ACIDS.

Place the flask on a water bath and evaporate the alcohol. Add such an amount of half-normal hydrochloric acid that its volume plus the amount used in titrating for the saponification number will be 1 cubic centimeter in excess of the amount required to neutralize the 50 cc of alcoholic potash added. Connect the flask with a condensing tube 3 feet long made of small glass tubing and place it on the steam bath until the separated fatty acids form a clear stratum on the upper surface of the liquid. Fill the flask to the neck with hot water and cool it in ice water until the cake of fatty acids is thoroughly hardened. Pour the liquid contents of the flask through a dry weighed filter into a liter flask, taking care not to break the cake. Fill the flask again with hot water, set on steam bath until the fatty acids collect at the surface, cool by immersing in ice water, and filter the liquid again into the liter flask. Repeat this treatment with hot water, followed by cooling and filtration of the wash water three times, collecting the washings in the liter flask, and titrate with deci-normal alkali, using phenolphthalein as indicator.

The number of cubic centimeters of deci-normal alkali used in this titration diminished by 5 (corresponding to the excess of 1 cc of half-normal acid) and multiplied by 0.0088 gives the weight of butyric acid in the amount of fat saponified; dividing this by the weight of fat taken gives the percentage of soluble acids.

(e) INSOLUBLE ACIDS OR HEHNER NUMBER.

Allow the flask containing the cake of insoluble acids and the filter paper through which the soluble acids have been filtered to drain and dry for twelve hours in the air. Transfer the filter paper to the flask and dry the flask and contents for three hours in a water-jacketed oven, cool, and weigh. Then dry for another two hours, cool, and weigh. If there be any considerable decrease in weight, repeat the drying. The weight obtained less the weight of the filter paper gives weight of insoluble acids, from which the percentage can be easily calculated.

6.—DETERMINATION OF FREE FATTY ACIDS.<sup>b</sup>

Weigh 20 grams of fat or oil into a flask, add 50 cc of 95 per cent alcohol which has been neutralized with weak caustic soda, using phenolphthalein as indicator, and heat to boiling point. Agitate the flask thoroughly in order to dissolve the free fatty acids as completely as possible. Titrate with deci-normal alkali, agitating thoroughly until the pink color persists after vigorous shaking.

Express results either as percentage of oleic acid, as acid degree (cubic centimeters of normal alkali required to neutralize the free acids in 100 grams of oil or fat), or as acid value (milligrams of potassium hydroxid required to saturate the free acids in 1 gram of fat or oil).

1 cc deci-normal alkali = 0.0282 grams oleic acid.

7.—DETERMINATION OF VOLATILE ACIDS OR REICHERT-MEISSL NUMBER.

See methods for dairy products p. 38.

<sup>a</sup> Allen, *Com. Org. Anal.*, 3d ed., vol. 2, pt. 1, pp. 53-55.

<sup>b</sup> Allen, *Com. Org. Anal.*, 3d ed., vol. 2, p. 105.

8.—FOR ESTIMATION OF LIQUID AND SOLID FATTY ACIDS, MUTER'S METHOD<sup>a</sup> MODIFIED BY LANE.<sup>b</sup>

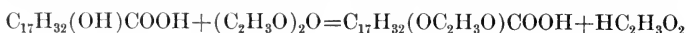
Weigh 5 grams of oil or fat into an Erlenmeyer flask, saponify, precipitate with lead acetate, and extract with ether, as directed under determination of arachidic acid. Filter the ether solution of soluble lead soap into a Muter tube or separatory funnel and decompose the soap by shaking with 40 cc of a 1:5 solution of hydrochloric acid. The soap is completely decomposed when the ether becomes clear and colorless.

The lead chlorid is drawn off from the ether solution and the ether washed free from acid. An aliquot of this ether solution is evaporated free from ether in an atmosphere of carbon dioxide in order to prevent the oxidation of the oleic acid, and weighed to determine the per cent of liquid acids; .2 to .3 gram of this is weighed and the iodine number determined in the ordinary way.

As it is very difficult to dry the oleic acid without very serious oxidation, it is just as satisfactory to determine the weight of insoluble acids by the following method: Wash the insoluble soap left on the filter into a flask, decompose with hydrochloric acid, and heat until the fatty acids are melted. Fill the flask with hot water, cool, pour off the water, and wash again the solidified fatty acids. Dissolve them in hot 95 per cent alcohol, transfer to weighed dish, remove the alcohol by evaporation, dry, weigh, and calculate the percentage of solid fatty acids.

9.—DETERMINATION OF ACETYL VALUE.<sup>c</sup>

Benedikt proposed to determine the hydroxy acids and alcohols by the use of acetic anhydrid  $(C_2H_3O)_2O$  as illustrated in the following reaction:<sup>d</sup>



He proposed to work on the fatty acids, but the process was modified by Lewkowitsch<sup>e</sup> who works on the oils or fats directly, which gives more exactly the true content of hydroxy acids.<sup>f</sup>

The procedure is as follows:

Boil the oil or fat with an equal volume of acetic anhydrid  $(C_2H_3O)_2O$  for two hours and pour the mixture into a large beaker containing 500 cc of water and boil for half an hour. To prevent bumping, a slow current of carbonic acid is passed into the liquid through a finely drawn out tube reaching nearly to the bottom. Allow the mixture to separate into two layers, siphon off the water, and boil the oily layer with fresh water until it is no longer acid to litmus paper.

The acetylated fat is then separated from the water and dried and filtered in a drying oven.

Weigh from 2 to 4 grams of the acetylated fats into a flask and saponify with alcoholic potash as in the determination of saponification equivalent. If the distillation process is to be adopted it is not necessary to work with a standardized alcoholic potash solution. In case the filtration method is used, which will be found much shorter, it is necessary that the alcoholic potash should be measured exactly.

In either case evaporate the alcohol after saponification and dissolve the soap in water. Now two procedures are possible—either distillation or filtration.

(a) DISTILLATION PROCESS.

Acidify with dilute sulphuric acid (1-10) and distill the liquid as in the Reichert test. As several hundred cubic centimeters must be distilled, either a current of

<sup>a</sup>J. Muter and L. L. De Koningh, *Analyst*, 1889, **14**, 61.

<sup>b</sup>N. J. Lane, *Jour. Am. Chem. Soc.*, 1893, **15**, 110.

<sup>c</sup>Lewkowitsch, *Jour. Soc. Chem. Ind.*, 1897, **16**, 503-506; Benedikt, *Analyse der Fette u. Wach.*, 3d ed., p. 146; Allen, *Com. Org. Anal.*, 3d ed., 2, pt. 1, pp. 66-67.

<sup>d</sup>Benedikt and Lewkowitsch, *Oils, Fats, and Waxes*, p. 127.

<sup>e</sup>*Jour. Soc. Chem. Ind.*, 1897, **16**, 503.

<sup>f</sup>J. Lewkowitsch, *Jour. Soc. Chem. Ind.*, 1890, **9**, 846.

steam is run through or portions of water are added from time to time. From 500 to 700 cc of distillate will be found to be sufficient. Filter the distillates to remove any insoluble acids carried over by the steam, and titrate the filtrate with deci-normal potassium hydroxid, using phenolphthalein as indicator. Multiply the number of cubic centimeters of alkali employed by 5.61 and divide by the weight of substance taken. This gives the acetyl value.

#### (b) FILTRATION PROCESS.

Add to the soap solution a quantity of standard sulphuric acid exactly corresponding to the amount of alcoholic potash added, warm gently, and the free fatty acids will collect on top.

Filter off the liberated fatty acids, wash with boiling water until the washings are no longer acid, and titrate the filtrate with deci-normal potassium hydroxid, using phenolphthalein as indicator. Calculate the acetyl value as before.

#### 10.—DETERMINATION OF PHYTOSTEROL AND CHOLESTEROL.<sup>a</sup>

Boil 50 grams of fat or oil in a flask with reflux condenser with 75 cc of 95 per cent alcohol for five minutes and separate alcoholic solution. Repeat with another portion of alcohol and separate. Mix the alcoholic solution with 15 cc of 30 per cent sodium hydroxid and boil in a flask with a condensation tube until one-fourth of the alcohol is evaporated. Evaporate nearly to dryness in porcelain dish and shake the residue with ether. The ethereal solution is evaporated to dryness, taken up with a little ether, filtered, again evaporated, dissolved in hot 95 per cent alcohol and allowed to crystallize.

Cholesterol can easily be distinguished from phytosterol by the form and grouping of the crystals; also by the melting point, which is 146° C.,<sup>b</sup> while that of phytosterol is from 130° to 137.5° C.<sup>c</sup>

Phytosterol is found in most vegetable oils, with the notable exception of olive and palm oil. The crystals as separated from hot alcohol appear in tufts of needles.

Cholesterol is characteristic of animal fats. It crystallizes in thin rhombic tables.

#### 11.—DETERMINATION OF THE UNSAPONIFIABLE RESIDUE.<sup>d</sup>

Saponify 5 grams<sup>e</sup> of oil or fat with alcoholic potassium hydroxid and remove the alcohol by evaporation. Wash into separatory funnel with from 70 to 100 cc of water and extract with from 50 to 60 cc of ether. In case the two liquids do not separate, a few cubic centimeters of alcohol may be added. Separate the water solution and wash the ether with water containing a few drops of sodium hydroxid. Again extract the soap solution and washings with ether and evaporate the combined extracts to dryness. In most cases it is advisable to add a little alcoholic potassium hydroxid to the residue and heat in order to saponify any traces of fats left unsaponified and extract again with ether. Transfer to a weighed dish and dry as quickly as possible in a water oven.

Many of the hydrocarbon oils are volatile at 100° C., so that the drying should not be carried any further than necessary. With resin oil, paraffin wax, and the denser mineral oils there is little danger of loss at 100°.

On account of the solubility of soap in ether and petroleum ether it is well to wash the residue with warm water containing a little phenolphthalein. If it shows alkaline reaction there is soap present.

<sup>a</sup>Forster and Reichelmann *Analyst*, 1897, **22**, 131; E. Salkowski, *Ztsch. anal. Chem.*, 1887, **26**, 557; E. Von Raumer, *Ztsch. angew. Chem.*, 1898, **13**, 555-556; *Jour. Soc. Chem. Ind.*, 1898, **17**, 774; H. Kreis and O. Wolf, *Jour. Soc. Chem. Ind.*, 1898, **17**, 1075.

<sup>b</sup>E. Salkowski, *Ztschr. anal. Chem.*, 1887, **26**, 557.

<sup>c</sup>Bömer, *Ztschr. Unter. d. Nahr u. Genuss*, 1898, **1**, 81.

<sup>d</sup>Allen, *Com. Org. Anal.*, 3d Ed., Vol. 2, pp. 1 and 113.

<sup>e</sup>See Appendix, p. 150.

12.—DETERMINATION OF MELTING POINTS OF FATS<sup>a</sup>—WILEY'S METHOD.<sup>b</sup>

## (a) PREPARATION OF REAGENTS.

Have a piece of ice floating in distilled water that has been recently boiled. Prepare a mixture of alcohol and water of the same specific gravity as the fat to be examined. This is done by boiling distilled water and 95 per cent alcohol for ten minutes to remove the gases which they may hold in solution. While still hot, the water is poured into the test tube described below (2) until it is nearly half full. The test tube is nearly filled with the hot alcohol, which is carefully poured

down the side of the inclined tube to avoid too much mixing. If the alcohol is not added until the water has cooled, the mixture will contain so many air bubbles as to be unfit for use. These bubbles will gather on the disk of fat as the temperature rises and finally force it to the top.

## (b) APPARATUS.

The apparatus for determining the melting point consists of an accurate thermometer reading easily tenths of a degree; a cathetometer for reading the thermometer (but this may be done with an eyeglass if held steadily and properly adjusted); an ordinary thermometer; a tall beaker 35 cm high and 10 cm in diameter; a test tube 30 cm long and 3.5 cm in diameter; a stand for supporting the apparatus; some method of stirring the water in the beaker (for example, a blowing bulb of rubber, and a bent glass tube extending to near the bottom of the beaker). (See fig. 2.)

## (c) DETERMINATION.

The disks of fat are prepared as follows: The melted and filtered fat is allowed to fall from

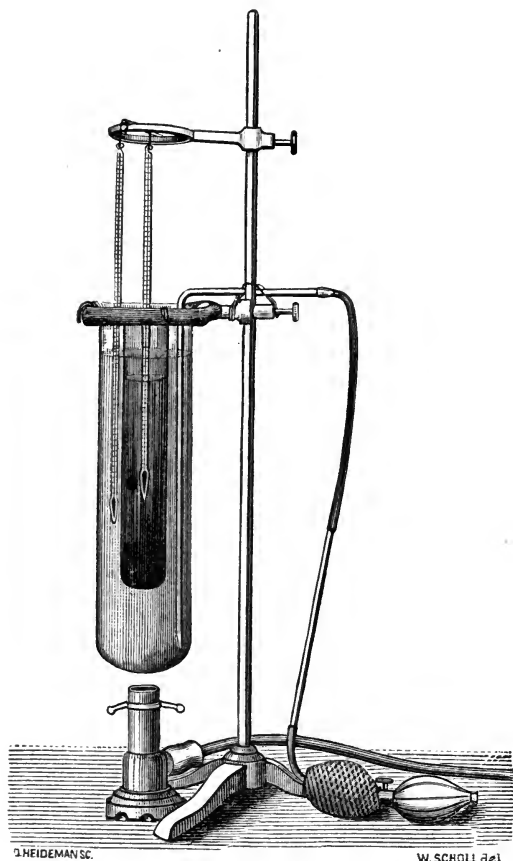


FIG. 2.—Apparatus for the determination of the melting point.

a dropping tube from a height of from 15 to 20 cm on a smooth piece of ice floating in distilled water that has been recently boiled. The disks thus formed are from 1 to 1.5 cm in diameter, and weigh about 200 mg. By pressing the ice under the water the disks are made to float on the surface, whence they are easily removed with a steel spatula, which should be cooled in the ice water before using.

The disks must be allowed to stand for two or three hours in order to obtain the normal melting point.

The test tube containing the alcohol and water is placed in a tall beaker contain-

<sup>a</sup> See Appendix, p. 151.

<sup>b</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 52.

ing water and ice, until cold. The disk of fat is then dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the alcohol water is exactly equivalent to its own. Here it remains at rest and free from the action of any force save that inherent in its own molecules.

The delicate thermometer is placed in the test tube and lowered until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disk, the thermometer is moved from time to time in a circularly pendulous manner.

The disk having been placed in position, the water in the beaker is slowly heated and kept constantly stirred by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about  $6^{\circ}$  C. below the melting point, the disk of fat begins to shrivel and gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotary movement should be given to the thermometer bulb. The rise of temperature should be so regulated that the last  $2^{\circ}$  C. of the increment require about ten minutes. The mass of fat gradually approaches the form of a sphere, and, when it is sensibly so, the reading of the thermometer is to be made. As soon as the temperature is taken the tube is removed from the bath and placed again in the cooler. A second tube, containing alcohol and water, is at once placed in the bath. The test tube (ice water having been used as a cooler) is of low enough temperature to cool the bath sufficiently. After the first determination, which should be only a trial, the temperature of the bath should be so regulated as to reach a maximum of about  $1.5^{\circ}$  above the melting point of the fat under examination.

The edge of the disk should not be allowed to touch the sides of the tube. This accident rarely happens, but in case it should take place and the disk adhere to the sides of the tube a new trial should be made.

Triplicate determinations should be made, and the second and third results should show a near agreement.

Example: Melting point of sample of butter:

	Degrees.
First trial .....	33.15 C.
Second trial .....	33.05 C.
Third trial .....	33.10 C.

### 13.—DETERMINATION OF MELTING POINT OF FATTY ACIDS.<sup>a</sup>

Draw up the melted fatty acid into a very thin-walled capillary tube 1 or 2 inches long according to the length of bulb of the thermometer used. Seal one end of the tube and allow the fatty acid to cool on ice for from twelve to fifteen hours. Then attach to the bulb of a delicate thermometer graduated to one-fifth degree, immerse in a beaker of water, and warm up very slowly. The point where the acid becomes transparent is taken as the melting point.

### 14.—DETERMINATION OF MAUMENÉ NUMBER.<sup>b</sup>

The following apparatus has been largely used by the writer and has given very satisfactory results:

A beaker, 5 inches by  $1\frac{1}{2}$  inches, is placed inside of another 6 inches by 3 inches, and a wet mixture of asbestos and plaster of paris tightly packed around the inner beaker. This, when dried, makes a hard, solid packing which radiates heat very slowly.

<sup>a</sup>U. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 4, p. 448. Benedikt and Lewkowitsch, *Oils, Fats, and Waxes*, p. 97. Wiley, Prin. and Prac. Agr. Anal., vol. 3, p. 321.

<sup>b</sup>Allen, *Com. Org. Anal.*, 3d ed., vol. 2, pt. 1, p. 76.

Remove the inner beaker, weigh into it 50 grams of fat, and note the temperature carefully. Then, from a pipette which will deliver it in approximately one minute, add 10 cc of the strongest sulphuric acid<sup>a</sup> which is at the same temperature as the oil.

While the acid is being introduced, stir the oil and acid with an accurate thermometer. Then hold the thermometer bulb carefully in the center of the mixture, and when the mercury reaches the highest point note the reading. It is easy to determine this point as the column of mercury remains stationary for some time. It is necessary to take care not to read the temperature too soon, as some oils take considerable time to reach their maximum point.

The difference between the initial reading and the final reading, expressed in degrees centigrade, gives the Maumené number.

Great care must be taken to have the acid of the highest strength. With the semi-drying oils, such as cotton-seed, the use of this strength of acid will cause foaming and make it almost impossible to obtain the correct rise of temperature. With such oils, either a weaker acid will have to be used and the results compared with the rise of temperature with water, or a dilution with paraffin oil made. It is always best to test the apparatus and acid by use of water and oils of known purity. With 50 grams of water and 10 cc of 99 per cent sulphuric acid, Thomson and Ballantyne<sup>b</sup> obtained a rise of 46.5° C. Working with acid of specific gravity of 1.844, the average of a number of determinations in this laboratory was 45°, but this will vary with the apparatus and manipulator.

The acid which is used in testing the apparatus should be used in all the determinations and care should be taken that it does not lose its strength. When this test is conducted with care, it is one of the most valuable in detection of adulteration in fats and oils.

In reporting results obtained, the rise of temperature with water should be stated, otherwise no comparative value can be attached to the results.

#### 15.—DETERMINATION OF RESIN OIL.

Take the pure oil or a definite dilution with petroleum ether and polarize in a 200 mm tube.

Resin oil has a polarization of from +30 to +40 on the sugar scale (Schmidt and Haensch) in a 200 mm tube while other oils read between 1°+ and -1°.

#### 16.—HALPHEN<sup>d</sup> REACTION FOR COTTON-SEED OIL.

Carbon disulphid, containing about 1 per cent of sulphur in solution, is mixed with an equal volume of amyl alcohol. Mix equal volumes of this reagent and the oil under examination and heat in a bath of boiling brine for fifteen minutes. In the presence of as little as 1 per cent of cotton-seed oil, an orange or red color is produced, which is characteristic.

Lard and lard oil from animals fed on cotton-seed meal will give a faint reaction; also the fatty acids.

This test is more sensitive than the Bechi test and less liable to give unsatisfactory results in the hands of an inexperienced person. It is not affected by rancidity. The depth of color is proportional, to a certain extent, to the amount of oil present, and by making comparative tests with cotton-seed oil some idea as to the amount present can be obtained, but it must be remembered that different oils react with different intensities, and oils which have been heated to 200° to 210° C. <sup>d</sup>react with greatly

<sup>a</sup> See appendix, p. 151.

<sup>b</sup> Jour. Soc. Chem. Ind., 1891, 10, 234.

<sup>c</sup> G. Halphen, Jour. Pharm. Chim., 1897, 6, 390-391. Analyst, 1897, 22, 326. Allen, Com. Org. Anal., 3d ed., vol. 2, pt. 1, p. 143. Winton, Conn., Exp. Sta. Rept., 1900, pt. 2, p. 144.

<sup>d</sup> Allen, Com. Org. Anal., 3 ed., vol. 2, pt. 1, p. 143.



diminished intensity. Heating ten minutes at 250° renders cotton-seed oil incapable of giving reaction.<sup>a</sup>

#### 17.—BECHI OR SILVER NITRATE TEST FOR COTTON-SEED OIL.<sup>b</sup>

*Reagent.* <sup>c</sup>—Dissolve 2 grams of silver nitrate in 200 cc of 95 per cent alcohol and 40 cc of ether, adding 1 drop of nitric acid.

Mix 10 cc of oil or melted fat, 5 cc of reagent, and 10 cc of amyl alcohol<sup>d</sup> in a test tube. Divide, heat one-half in a boiling water bath for ten minutes, and then compare with portion not heated. Any blackening due to reduced silver shows presence of cotton-seed oil.

Other oils which have become rancid,<sup>e</sup> and lards which have been steamed or heated at high temperature, contain decomposition products which have a reducing action on silver nitrate. The writer found in testing a large number of salad oils some which contained no cotton-seed oil, according to the Halphen test, but gave a brown coloration with Bechi reagent, and in some cases reduced silver. These same oils on being purified gave no reaction. Hence the oils or fats should be purified before testing.

To purify the oils and fats, heat from 20 to 30 grams on water bath for a few minutes with the addition of 25 cc of 95 per cent alcohol, <sup>f</sup> shake thoroughly, decant as much of the alcohol as possible, and wash with 2 per cent nitric acid,<sup>g</sup> and finally with water. The oil or lard thus purified will give no reduction at all if it contains no cotton-seed oil. Heating the oils or fats to 100° C. or simple washing with 2 per cent nitric acid is not sufficient except in a few cases.

With oils the use of the Halphen and Bechi tests will be found to be useful as a means of approximately determining the amounts of adulteration present. If Halphen gives a reaction and Bechi does not, the adulteration with cotton-seed oil is probably less than 20 per cent.

#### 18.—RENARD'S<sup>h</sup> TEST FOR PEANUT OIL AS MODIFIED BY TOLMAN.

Weigh 5 grams of oil into an Erlenmeyer flask, as directed under determination of saponification number. (If this is accurately weighed and a standard solution of alcoholic potash used, the saponification number can be determined on the same sample by titrating the excess of alcoholic potash used in the saponification with half-normal acetic acid.) Saponify with alcoholic potash, naturalize exactly with dilute acetic acid, using phenolphthalein as indicator, and wash into a 500-cc flask containing a boiling mixture of 200 cc of water, and 60 cc of a 10 per cent lead acetate solution. Boil for a minute, and then cool the precipitated soap by immersing the flask in water, occasionally giving the flask a whirling motion to cause the soap to stick to the sides of the flask. After the soap has cooled, the water and excess of lead can be poured off, and the soap washed with cold water and with 90 per cent (by volume) alcohol.<sup>i</sup> Now, add 200 cc of ether, cork the flask, and allow to stand for some time until the soap is disintegrated, then heat on the water bath, using a reflux condenser, and boil for about five minutes. In the oils most of the soap will

<sup>a</sup> D. Holde and R. Pelgry, *Jour. Soc. Chem. Ind.*, 1899, **18**, 711.

<sup>b</sup> See appendix, p. 151.

<sup>c</sup> Pearmain and Moor, *Allen Com. Org. Anal.*, 3 ed., vol. 2, pt. 1, p. 143. Wesson, *Jour. Am. Chem. Soc.*, 1895, **17**, 724.

<sup>d</sup> The addition of amyl alcohol is not necessary, but the writer finds it very convenient, as it dissolves the oils or fats and enables one to mix the oil and reagent much better.

<sup>e</sup> Wesson, *Jour. Am. Chem. Soc.*, 1895, **17**, 724. A. L. Winton, *Conn. Expt. Sta. Rept.*, 1900, pt. 2, p. 143.

<sup>f</sup> Used by the writer and found to be much more convenient and just as satisfactory as dilute alkali.

<sup>g</sup> Wesson, *Jour. Am. Chem. Soc.*, 1895, **17**, 724.

<sup>h</sup> Renard, *Comp. Rend.*, 1871, **73**, 1330. Benedikt and Lewkowitsch, *Oils, Fats, and Waxes*, p. 365.

<sup>i</sup> Process used by N. J. Lane in his modification of Muter's method. *Jour. Am. Chem. Soc.*, 1893, **15**, 110.

be dissolved, while in lards, where there is so much stearin, part will be left undissolved. Cool the ether solution of soap down to from 15° to 17° C., and allow to stand until all the insoluble soaps have crystallized out. It should stand about twelve hours.

Now, filter and wash the precipitate with ether. Save the filtrate for the determination of the iodine number of the liquid fatty acids by the Muter method.

The soaps on the filter are washed back into the flask by means of a stream of hot water acidified with hydrochloric acid.

Add an excess of dilute hydrochloric acid, fill up the flask with hot water, allow the free fatty acids to harden and separate from the precipitated lead chlorid, wash, drain, and dissolve the fatty acids in 25 cc of boiling 90 per cent (by volume) alcohol. The crystals of arachidic acid separate out as the liquid cools. From 5 to 10 per cent of peanut oil can be detected by this method, as it effects a complete separation of the soluble acids from the insoluble, which interfere with the crystallization of the arachidic acid. Filter, wash the precipitate twice with 10 cc of 90 per cent (by volume) alcohol, and then with alcohol of 70 per cent (by volume). Dissolve off the filter with boiling absolute alcohol, evaporate to dryness in a weighed dish, dry and weigh. Add to this weight 0.0025 gram for each 10 cc of 90 per cent alcohol used in the crystallization and washing if done at 15° C., if done at 20°, 0.0045 gram for each 10 cc.

The melting point of arachidic acid obtained in this way is between 71° and 72° C. Twenty times the weight of arachidic acid will give the approximate amount of peanut oil present.

Another method<sup>a</sup> which gives as satisfactory an approximation of the amount of peanut oil present is to allow the arachidic<sup>b</sup> acid to crystallize in a 100 cc graduated cylinder and measuring the volume of the precipitate. This volume will have to be determined for the working temperature and the length of the time by use of known mixtures of peanut oil. Cotton-seed and lard oil give slight precipitates when treated by this method.

Arachidic acid has a characteristic structure and can be detected by the microscope.

No examination of olive oil is complete without making the test for peanut oil, which is probably a common adulterant, especially in French and Italian oils.

#### 19.—BAUDOUIN TEST FOR SESAME OIL.

Dissolve 0.1 gram of finely powdered sugar in 10 cc of hydrochloric acid (sp. gr. 1.20), add 20 cc of the oil to be tested, shake thoroughly for a minute and allow to stand. The aqueous solution separates almost at once. In the presence of even a very small admixture of sesame oil, this is colored crimson. Some olive oils give a slight pink coloration with this reagent, but they are not hard to distinguish if comparative tests with sesame oil are made.

#### 20.—VILLIVECCHIA<sup>c</sup> TEST FOR SESAME OIL.

Mix 2 grams of furfural with 100 cc alcohol (95 per cent), and take 0.1 cc of this solution, 10 cc hydrochloric acid (sp. gr. 1.20), and 10 cc of oil and mix thoroughly by shaking in a test tube and the same color is developed as when the sugar is used. Villivecchia attributed the Baudouin test to the formation of furfural from the action of levulose and hydrochloric acid, and so substituted furfural for sucrose.

As furfural and hydrochloric acid give a violet tint with hydrochloric acid, it is necessary to use the very dilute solution given in the method.

<sup>a</sup> Suggested by W. D. Bigelow.

<sup>b</sup> As the solubility of arachidic acid in 90 per cent alcohol increases very rapidly with the temperature, care must be taken to keep the temperature of crystallization down to between 15° and 20° C., and to obtain satisfactory results the temperature must be same as used in the standards.

<sup>c</sup> Villivecchia and Fabris, *Journ. Soc. Chem. Ind.*, 1893, **12**, 97 and 1894, **13**, 69. Benedikt and Lewkowitzsch, *Oils, Fats, and Waxes*, p. 318.

21.—TOCHER<sup>a</sup> TEST FOR SESAME OIL.

Dissolve 1 gram pyrogallol in 15 cc of concentrated hydrochloric acid. Mix this solution with 15 cc of oil in a separatory funnel and allow to stand for a minute. Draw off the aqueous layer and boil for five minutes. In the presence of sesame oil it becomes colored red by transmitted light and blue by reflected light.

22.—MICROSCOPICAL EXAMINATION.<sup>b</sup>

Dissolve in a test tube from 2 to 5 grams of oil or fat in about 10 cc of ether, plug the test tube lightly with cotton and allow to stand 15 or more hours in a moderately cool place.

The most characteristic crystals are obtained when the crystallization proceeds slowly and at temperature of from 22° to 24° C. The first crop of crystals may be examined and the mother liquor separated and set aside for further crystallization.

In order to get rid of the oleins, Gladding<sup>c</sup> has suggested the following:

Dissolve in an Erlenmeyer flask 5 grams of melted fat in 10 cc of absolute alcohol and 5 cc of ether, stopper with cotton and place in ice water for about one-half hour, until the more crystallizable portions of the fat have separated. The crystalline part is separated by filtration through a filter paper moistened with alcohol, and washed with the alcohol-ether mixture. After drying in the air for some time the crystals are dissolved from the paper by means of ether and then treated in the same way as described in the first method. When the crystals are ready to examine a drop is removed with a pipette, placed on a slide, a drop of cotton oil or olive oil added, and a cover slip pressed gently down.

## III.—DAIRY PRODUCTS.

By J. A. LECLERC,

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## (A) MILK AND CREAM.

## 1.—GENERAL DISCUSSION.

There are three kinds of adulteration generally practiced with milk. First, addition of water, which is the simplest and the most common practice. Second, removal of fat or the removal of fat and addition of water. This double adulteration is used in order not to disturb the specific gravity. Third, the addition of preservatives, most commonly formaldehyde, boric acid, or borax.

The determinations ordinarily made in the examination of milk and cream are specific gravity, fat, total solids, solids not fat, and the detection of preservatives and coloring matter. The specific gravity alone is of little value in judging the purity of milk, owing to the fact that the increase of specific gravity produced by the removal of cream may be reduced by the addition of water. One of the most important considerations is the relation of the solids not fat to the fat. In milk it has been found that this ratio does not vary widely from 9:4.

2.—DETERMINATION OF TOTAL SOLIDS.<sup>d</sup>

Heat at 100° C. to constant weight about 3 grams of milk in a tared platinum, aluminum, or tin dish<sup>e</sup> of 5 cm diameter, with or without the addition of 15 to 30 grams of sand. Cool and weigh.

<sup>a</sup>Pharm. Journ. and Trans., 1891, 639. Chem. Zeit., Rep., 1891, 5, 15-33. Benedikt and Lewkowitsch, Oils, Fats, and Waxes, p. 319. Winton, Conn. Expt. Sta. Rept., 1900, pt. 2, p. 153.

<sup>b</sup>U. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 4, p. 449. Gladding, Jour. Am. Chem. Soc., 1896, 18, 189. Wiley Prin. & Prac. Agri. Anal., vol. 3, pp. 345, 346. Winton, Report Conn. Expt. Sta., 1900, pt. 2, p. 145.

<sup>c</sup>Jour. Am. Chem. Soc., 1896, 18, 189.

<sup>d</sup>U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 54.

<sup>e</sup>See Appendix, p. 151.

## 3.—DETERMINATION OF FAT.

(a) OFFICIAL METHOD.<sup>a</sup>

Dry about 5 grams of the sample on ignited asbestos in a Hofmeister schälchen or in a perforated metal cylinder (described by Babcock) and extract with ether in a continuous extraction apparatus.

(b) BABCOCK'S METHOD.<sup>b</sup>

This method is commonly used where a large number of samples is to be examined. Owing to the general use of this method and its wide publication it is not deemed advisable to introduce its description here.

## (c) GERBER'S METHOD.

Where only occasional samples are to be examined Gerber's acid butyrometer is found to give results comparable with those of the Babcock apparatus and is much more convenient. Directions accompany the apparatus.

## 4.—DETERMINATION OF SOLIDS NOT FAT.

Deduct the percentage of fat from the percentage of total solids.

5.—DETECTION OF GELATIN.<sup>c</sup>

"An acid solution of mercuric nitrate is prepared by dissolving mercury in twice its weight of nitric acid of 1.42 specific gravity, and diluting this solution to 25 times its bulk by the addition of water. Ten cubic centimeters of the milk or cream to be examined are mixed with an equal volume of the acid mercuric nitrate solution, the mixture is shaken, and then 20 cc of water are added. The liquid is again shaken, allowed to stand five minutes, and filtered. In the presence of much gelatin the filtrate will be opalescent and can not be obtained quite clear. To a portion of the filtrate contained in a test tube an equal volume of a saturated aqueous solution of picric acid is added. A yellow precipitate will be produced in presence of any considerable amount of gelatin, while smaller amounts will be indicated by the cloudiness produced by the picric acid solution. In the absence of gelatin the filtrate obtained will be perfectly clear, and will be unaffected by adding picric acid."

6.—DETECTION OF FORMALDEHYDE.<sup>d</sup>

To the milk to be tested add strong commercial sulphuric acid without mixing, and at the junction of the two liquids a violet or blue color will appear if the milk contains one or more parts per 10,000 of formaldehyde. This color is supposed to be given only when there is a trace of ferric chlorid or other oxidizing agent present.

Other methods of detecting formaldehyde are described on pages 79 and 107.

## 7.—DETECTION OF BORAX AND BORIC ACID.

Use methods described on page 110.

## 8.—DETECTION OF FOREIGN COLORS.

(a) LEACH'S METHOD.<sup>e</sup>

Warm about 150 cc of milk in a casserole over the flame and add about 5 cc of acetic acid, after which slowly continue the heating nearly to the boiling point while

<sup>a</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 54.

<sup>b</sup> Wis. Exp. Sta. Bul. No. 24, and U. S. Dept. of Agr., Div. of Chem., Bul. No. 28, p. 34-42.

<sup>c</sup> Allen, Com. Org. Anal., 2d ed., Vol. IV, pp. 181-182.

<sup>d</sup> See Appendix, p. 151.

<sup>e</sup> Jour. Am. Chem. Soc., 1900, 22, 207.

stirring. Gather the curd, when possible, into one mass by the stirring rod, and pour off the whey. If the curd breaks up into small flecks, separate from the whey by straining through a sieve or colander. Press the curd free from adhering liquid, transfer to a small flask, and macerate for several hours (preferably over night) in about 50 cc of ether, the flask being tightly corked and shaken at intervals.

(1) *Detection of annatto (in the ether extract).*

Decant the ether extract into an evaporating dish, place on the water bath, and evaporate off the ether. Make the fatty residue alkaline with sodium hydroxid, and pour upon a very small wet filter while still warm. After the solution has passed through, wash off the fat from the filter with a stream of water and dry the paper. If, after drying, the paper is colored orange, the presence of annatto is indicated, confirmed by applying a drop of stannous chlorid solution, which, in presence of annatto, produces a characteristic pink on the orange-colored paper.

(2) *Detection of aniline orange (in the curd).*

The curd of an uncolored milk should be perfectly white after complete extraction with ether, as would also that of a milk colored with annatto.

If the extracted fat-free curd is distinctly dyed an orange or yellowish color, aniline orange is indicated. To confirm the presence of this color, treat a lump of the fat-free curd in a test tube with a little strong hydrochloric acid. If the curd immediately turns pink, the presence of aniline orange is assured.

(3) *Detection of caramel (in the curd).*

If the fat-free curd is colored a dull brown, caramel is to be suspected. Shake a lump of the curd, as in (b), with strong hydrochloric acid in a test tube and heat gently. The acid solution of the caramel-colored curd will gradually turn a deep blue, as would also the white, fat-free curd of an uncolored milk, while the curd itself does not change color.<sup>a</sup>

(b) **LYTHGOE'S TEST FOR ANILINE ORANGE.**<sup>b</sup>

Treat about 10 cc of the milk with an equal volume of hydrochloric acid (sp. gr. 1.20) in a porcelain casserole, and give the dish a slight rotary motion. If aniline orange is present in appreciable amount a pink color will at once be imparted to the curd particles as they separate out.

**(B) BUTTER.**

1.—GENERAL DISCUSSION.

The most common adulteration of butter is the substitution of fat other than butter fat. This is effected either by oleomargarine or by mixtures of oleomargarine and butter. Within the last few years a new product, called process or renovated butter, has been sold extensively as butter. The process of its manufacture is, briefly, as follows:

Poor and rancid butter is melted, the curd and brine are allowed to settle, the froth and scum skimmed off, after which the clear fat is drawn off and completely aerated so as to remove any unpleasant odors. Next, pure skimmed or whole milk is added and the mixture is thoroughly stirred so as to form a complete emulsion, and is

<sup>a</sup> It should be noted that it is only when this blue coloration of the acid occurs in connection with a brown-colored curd, which itself does not change color, that caramel is to be suspected, as distinguished from the pink coloration produced at once under similar conditions by aniline orange. It is to be regretted that there are no such definite confirmatory tests for caramel as there are for annatto and aniline orange. See 19th An. Rep. Mass. State Board of Health (1887), p. 183.

<sup>b</sup> Jour. Am. Chem. Soc., 1900, 22, 813.

finally sprayed into ice-cold water so as to give the product that granular appearance usually found in butter just from the churn. It is then treated and sold as butter. When this product, or its mixture with butter, is sold as butter it should be considered an adulteration. In addition to the special methods given below for the detection of process butter, the general methods described under Edible Fats and Oils must often be employed. Excessive amounts of water or of casein should be regarded as adulterations. Occasionally preservatives other than salt are added to butter, but they are generally the same as those found in milk.

## 2.—DETERMINATION OF WATER.<sup>a</sup>

Place from 1.5 to 2.5 grams of the sample in a flat-bottomed dish having a surface of at least 20 square centimeters, and dry to constant weight at the temperature of boiling water.

The use of clean, dry sand or asbestos with the butter is admissible, and is necessary if a dish with round bottom be employed.

## 3.—DETERMINATION OF FAT.<sup>a</sup>

### (a) DIRECT METHOD.

Dry the butter on asbestos or sand to determine the water, and extract the fat by anhydrous alcohol-free ether. Evaporate the ether from the extract, heat to constant weight at the temperature of boiling water, and weigh.

### (b) INDIRECT METHOD.

Dissolve the dry butter from the water determination in the same dish with absolute ether or with 76° C. petroleum ether. Then transfer the contents of the dish to a weighed Gooch crucible with the aid of a wash bottle filled with the solvent, and wash until free from fat. Heat the crucible and contents at the temperature of boiling water until the weight is constant, and calculate the weight of fat from the data obtained.

## 4.—DETERMINATION OF REICHERT-MEISSL NUMBER.

Employ the official method of the association.<sup>b</sup>

## 5.—DETERMINATION OF SAPONIFICATION VALUE.

Proceed as directed on page 26.

## 6.—THE WATERHOUSE TEST FOR OLEOMARGARINE.<sup>c</sup>

Half fill a 100-cc beaker with sweet milk, heat nearly to boiling, and add 5 to 10 grams of butter or oleomargarine. Stir with a small rod, preferably of wood and about the size of a match, until the fat is melted. Then place the beaker in cold water and stir the milk until the temperature falls sufficiently for the fat to congeal. At this point the fat, if oleo, can easily be collected into one lump by means of the rod, while if butter it will granulate and can not be so collected. The distinction is very marked.

## 7.—SPECIAL TESTS FOR PROCESS BUTTER.<sup>d</sup>

### (a) FOAM TEST.<sup>e</sup>

Heat 2 or 3 grams of the sample, either in a spoon or dish, over a free flame. True butter will foam abundantly, whereas process butter will bump and sputter,

<sup>a</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 43.

<sup>b</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, pp. 44-46.

<sup>c</sup> Jour. Am. Chem. Soc., 1901, 23, 200; U. S. Dept. Agr., Farmers' Bul. 131, p. 7.

<sup>d</sup> See Appendix, p. 152.

<sup>e</sup> Jour. Amer. Chem. Soc., 1900, 22, 150; U. S. Dept. of Agr., Farmers' Bul. No. 131.

like hot grease, without foaming. Oleo behaves like process butter, but chemical tests and the Waterhouse test described above will indicate whether the sample is oleo or butter, either genuine or process.

(b) APPEARANCE OF MELTED BUTTER.<sup>a</sup>

Melt from 50 to 100 grams of butter or process butter at 50° C. The curd from the butter will settle, leaving above it a clear, supernatant fat. On the other hand the supernatant fat in the case of process butter does not assume that clear appearance, but remains more or less turbid.

(c) MICROSCOPIC EXAMINATION.<sup>b</sup>

Place a bit of the butter or process butter on a glass slide, cover it and press into a thin film with cover glass. Examine immediately with a polarizing microscope magnifying from 100 to 140 diameters. When a selenite plate is placed between the slide and the lower nicol a normal butter will give a uniformly blue colored field, showing the absence of fat crystals. On the other hand, process butter gives a blue field, mottled with yellow.

8.—DETECTION OF ANNATO AND SAFFRON IN BUTTER—CORNWALL'S METHOD.<sup>c</sup>

Five grams fat are dissolved in 50 cc of ether in a wide tube and the solution is vigorously shaken with 12 to 15 cc of a very dilute solution of potassium hydroxid, which must still be alkaline after it separates from the ether solution. It is allowed to stand a few hours, when the aqueous layer is drawn off, evaporated to dryness, and tested with sulphuric acid, which in the presence of annato gives first a blue or violet blue, changing quickly to green, and finally to brown.

Saffron which would be extracted at the same time acts differently when treated with sulphuric acid, not giving the green coloration.

The aqueous solutions, if not clear enough to use, must not be filtered, as the filter paper will take up large amounts of the color, but can be shaken up again with fresh portions of ether.

Martin<sup>d</sup> uses carbon disulphid as a solvent instead of ether, which is just as satisfactory.

Genuine butters treated in this way give only a very slight trace of coloring matter.

9.—DETECTION OF ANILINE COLORS.<sup>e</sup>

Follow methods described under Coloring Matter (p. 111 and following).

(C) CHEESE.

1.—GENERAL DISCUSSION.

There are two kinds of adulterations practiced with cheese. First, the use of fat other than milk fat, producing a product called filled cheese. Second, the removal of varying amounts of fat, producing skim cheese. The liquefied fats of swine or cattle intimately mixed with skim milk produce filled cheese; therefore the chemical methods for the examination of the fat of filled cheese are the same as are used for the detection of adulterated butter. Skim cheese is made from milk from which part or the whole of the fat has been removed; therefore the determination of fat and nitrogenous compounds will give a good indication as to whether the sample under examination is skim cheese.

<sup>a</sup> Jour. Amer. Chem. Soc., 1900, 22, 327.

<sup>b</sup> Jour. Amer. Chem. Soc., 1900, 22, 327.

<sup>c</sup> Chem. News, vol. 1887, 55, 49; U. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 1, p. 28.

<sup>d</sup> Analyst, 1885, 10, 163.

<sup>e</sup> See Appendix, p. 152.

2.—SELECTION OF SAMPLE.<sup>a</sup>

When the cheese can be cut take a narrow wedge-shaped segment reaching from the outer edge to the center of the cheese. Cut this into strips and pass through a sausage-grinding machine three times. When the cheese can not be cut take the sample with a cheese trier. If only one plug can be obtained take it perpendicular to the surface of the cheese at a point one-third of the distance from the edge to the center and extending either entirely or only half way through it. When possible draw three plugs—one from the center, one from a point near the outer edge, and one from a point halfway between the other two. For inspection purposes reject the rind; but for investigations requiring the absolute amount of fat in the cheese include the rind in the sample. It is preferable to grind the plugs in a sausage machine, but when this is not done they are cut very fine and carefully mixed.

3.—SEPARATION OF FAT FOR EXAMINATION.<sup>b</sup>(a) FIRST METHOD.<sup>c</sup>

Cut about 300 grams of cheese into fragments the size of a pea. Treat with 700 cc of potassium hydroxid (50 grams per liter) at 20° C. in a large wide-necked flask, and promote the solution of casein by vigorous shaking. In from 5 to 10 minutes the casein will be dissolved and the fat will come to the surface in lumps. Collect the lumps of fat into as large a mass as possible by a gentle shaking to and fro. Pour cold water into the flask until the fat is driven up into the neck and remove it by means of a spoon. Wash the fat thus obtained with as little water as will remove the residue of the lye which it may contain. Experience has shown that the fat is not perceptibly attacked by the lye in this treatment. By this method the fat is practically all separated in a short time and is then easily prepared for chemical analysis by filtering and drying as directed in the official method.<sup>d</sup>

## (b) SECOND METHOD.

Grind the cheese by passing it through a meat-cutting machine. Transfer it to a large flask and pour warm water upon it, using 1 cc for every gram of cheese. Shake thoroughly and add sulphuric acid (sp. gr. 1.82 to 1.825) slowly and in small quantities, shaking after each addition of acid. The total amount of acid used should be the same as the amount of water used. Remove the fat, which separates after standing a few minutes, by means of a separatory funnel, wash it free from acid, filter, and dry.

4.—DETERMINATION OF WATER.<sup>e</sup>

Place from 2 to 5 grams of cheese in a weighed platinum dish containing a small quantity of porous material such as ignited asbestos or sand, to absorb the fat which may run out of the cheese. Heat in a water-jacketed bath for ten hours and weigh; the loss in weight is considered as water. Or, if preferred, place the dish in a desiccator over concentrated sulphuric acid and dry to constant weight. Renew the acid when the cheese has become nearly dry.

5.—DETERMINATION OF FAT.<sup>e</sup>

Cover the perforations in the bottom of the extraction tube with dry asbestos, and on this place a mixture containing equal parts of anhydrous copper sulphate and

<sup>a</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 55.

<sup>b</sup> See also Appendix, p. 152.

<sup>c</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 51.

<sup>d</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 44, 3 (a).

<sup>e</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 56.



pure dry sand to the depth of about 5 cm. packing loosely. Cover the upper surface of this material with a film of asbestos and place on it from 2 to 5 grams of the sample of cheese. Place the tube in a continuous extraction apparatus and treat for five hours with anhydrous ether. Remove the cheese and grind to a fine powder with pure sand in a mortar. Replace the mixed cheese and sand in the extraction tube, wash the mortar free of all matters with ether, add the washings to the tube, and continue the extraction for ten hours.

#### 6.—DETERMINATION OF NITROGENOUS COMPOUNDS.

Make a determination of nitrogen by the Kjeldahl or the Gunning method, using about 2 grams of cheese, and multiply the percentage of nitrogen found by 6.25.

### IV.—CEREAL PRODUCTS.

By A. MCGILL,

*Chemist of Inland Revenue Laboratory, Ottawa, Canada.*

It has been found impossible to prepare the report on this subject this year. The heading has been inserted here to preserve its proper order.

### V.—INFANT AND INVALID FOODS.

By H. W. WILEY,

*Chief of Bureau of Chemistry, United States Department of Agriculture.*

#### 1.—GENERAL DISCUSSION.

Under this head are included all prepared foods of every description, which are intended especially for the use of infants and invalids.

It is evident that foods for infants should be as nearly as possible similar in character to the natural food, viz, healthy human milk. All modified milk, of the cow and other animals, intended for infants, would be included in this class. If these milks be evaporated, they would differ from the original sample only in the loss of water, provided the evaporation be carried on in vacuum at low temperature. Many of the foods advertised for the use of infants, however, contain starch and other matters not usually found in healthy human milk. The number of foods advertised for the use of infants is legion. Many of them are "predigested," that is, they have the proteid matter reduced to the form of more or less soluble protein and the starchy matters converted more or less completely into soluble carbohydrates.

Under the head of infant and invalid foods should also be considered the various products in which nitrogenous bodies are the most important ingredients.

Infant foods may be divided into two classes: First, milk of cows and other animals modified to resemble more or less completely healthy human milk; and, second, the foods in which carbohydrates are the predominating element. In this connection it may be stated that preparations of this nature are ordinarily used with milk, and may be considered in a sense as substitutes for milk sugar.

The modification of milk consists as a rule in diminishing the proportion of the protein matter or casein and increasing that of milk sugar and fat. By chemical and other treatment, the ordinary average milk of the cow may thus be modified so as to resemble more or less completely the average healthy human milk. It is planned to examine a large number of infant and invalid foods during the coming year, and for that reason the writer had intended to defer his report until the completion of the work mentioned. So many inquiries have recently been received regarding this subject, however, that it was thought best to prepare for this bulletin a brief summary of methods to be employed with references to a previous publication of the writer's describing them.

## 2.—METHODS OF DETERMINATION.

Determinations in this case should consist of:

## (a) WATER.

The water can be determined in the usual manner by evaporating a small quantity of the material in a flat dish, so that the film of solid matter may not be too thick. This is preferably done in vacuo or in an atmosphere of inert gas. See *Principles and Practice of Agricultural Analysis*, vol. 3, pages 13 and following; also *Department of Agriculture, Division of Chemistry, Bulletin No. 46 revised*, pages 27 and 43.

## (b) ASH.

Burn the dried sample at a low red heat, preferably in a muffle. See *Principles and Practice of Agricultural Analysis*, vol. 3, pages 36 and following; also *Bulletin 46 revised*, page 23.

## (c) FAT.

Determine by one of the methods given in *Principles and Practice of Agricultural Analysis*, vol. 3, pages 480 and following, and *Bulletin 46 revised*, page 54.

## (d) SUGARS.

The lactose may be determined both by reduction of copper salts and by optical processes. See *Principles and Practice of Agricultural Analysis*, vol. 3, pages 275 and following; and *Bulletin 46 revised*, pages 40 and following.

## (e) ADDED SUCROSE.

Use method of Bigelow and McElroy, *Principles and Practice of Agricultural Analysis*, vol. 3, page 296; or that of Stokes and Bodmer, *Analyst*, 1885, 10, 62.

## (f) PROTEIN.

Use methods described in *Principles and Practice of Agricultural Analysis*, vol. 3, pages 504 and following, for total protein and separation of protein matters; also *Bulletin 46 revised*, pages 54 and 55.

## 3.—CONDENSED MILK.

Mix the entire contents of the can, transfer 250 grams to a liter flask, dissolve in water and make the solution up to the mark. The solution should then be treated for various constituents as under dairy products on aliquot parts of the contents of the flask.

## 4.—CARBOHYDRATE FOODS.

Another class of foods for infants and invalids, as intimated above, is chiefly composed of carbohydrate bodies. These foods should be examined microscopically to determine, if possible, the origin and character of the starch. The water and ash should be determined by the usual methods. The quantity of starch unchanged should be determined. See *Principles and Practice of Agricultural Analysis*, vol. 3, pages 201 and following; and *Bulletin 46 revised*, page 25.

## (a) DEXTRIN.

This can be determined in the solution of the bodies after the fermentation of other sugars. Dextrin can then be determined by its opticity or by precipitation with alcohol. See *Principles and Practice of Agricultural Analysis*, vol. 3, pages 287 and following.

## (b) DEXTROSE.

Determine dextrose by the methods given in Principles and Practice of Agricultural Analysis, vol. 3, pages 287 and following.

## (c) INVERT SUGAR.

See Principles and Practice of Agricultural Analysis, vol. 3, pages 161, 162, 257 and following.

## (d) MALTOSE.

For separation from dextrin and dextrose, see Principles and Practice of Agricultural Analysis, vol. 3, pages 287 and following; for estimation, see pages 165 and following.

The important point to be determined in these foods is the extent to which the so-called predigestion has been carried, and this is done by ascertaining the condition of the carbohydrate and proteid bodies. Attention should also be given to the nature of any ferments which have been employed in effecting the predigestion, or acids, if such have been used. Further, these foods should be examined for preservatives, which are sometimes added when the samples are in a liquid state, or are perishable in character.

## VI.—SACCHARINE PRODUCTS.

By ALBERT E. LEACH,

*Analyst of State Board of Health, Boston, Mass.*

## 1.—GENERAL DISCUSSION.

This class of food products, from their nature and composition, is so closely allied to sugar and starch (methods for which have been already so fully studied by this association) that little remains of a purely distinctive character in their examination. Being, furthermore, composed almost exclusively of carbohydrates, it is doubtful if, as a rule, much attention need be given to the determination of nitrogen or fatty constituents, which occur in very minute quantities only, and chiefly in chocolate and flavoring material, or in the eggs and butter that enter incidentally into the manufacture of confectionery.

## 2.—PREPARATION OF THE SAMPLE.

## (a) MOLASSES AND SIRUP.

Insure a homogenous mixture by stirring with a rod till any crystallized sugar is evenly distributed throughout the mass.

## (b) HONEY.

Treat the strained honey as in the case of molasses, 2 (a).

In the case of comb honey, cut across the top of the comb, if sealed, and separate completely from the comb by straining through a 40-mesh sieve.

If the honey has become wholly or in part solidified by crystallization, use a gentle heat on a closed water bath to restore it to fluid form.

## (c) CONFECTIONERY.

(1) *Products of practically uniform composition throughout.*

(a) *Lozenges and other pulverizable products.*—Grind in a mortar or mill to a fine powder.

(b) *Semiplastic, sirupy, or pasty products.*—Weigh 50 grams of the sample into a 250-cc graduated flask, mix thoroughly or dissolve, if soluble, in water and fill to the mark. Be sure that the solution is uniform, or, if insoluble material is present, that it is evenly mixed by shaking before taking aliquot parts for the various determinations.

(2) *Confectionery in layers or sections of different composition.*

When it is desired to examine the different portions separately they should be separated mechanically with a knife when possible, and treated as directed under (1).

(3) *Sugar-coated fruit, nuts, etc.*

In case of a saccharine coating enclosing fruit, nuts, or any less readily soluble material, dissolve or wash off the exterior coating in water, which may, if desired, be evaporated to dryness for weighing, and proceed as in (1).

(4) *Brandy drops and similar preparations.*

In case of a hard exterior coating enclosing a sirup or fluid which it is desired to examine, puncture the outer coating with a knife and pour out the fluid, using a sufficient number of the "drops" to yield enough fluid for examination (see page 49, sec. 14).

(5) *Candied or sugared fruits.*

Proceed as directed under Preserves and Canned Fruits, page 75.

3.—DETERMINATION OF TOTAL SOLIDS.

(a) MOLASSES, SIRUPS, AND HONEY.

(1) *By direct determination.*

Weigh 20 grams into a 100-cc graduated flask, dissolve in water and make up to the mark. Insure a uniform solution by shaking. Measure 10 cc of this solution into a tared platinum dish containing about 5 grams of freshly ignited, finely divided asbestos fiber, and dry to constant weight at 70° in vacuo or in a McGill oven (see footnote on page 76).

(2) *By calculation from specific gravity.\**

Weigh 25 grams of the sample into a 100-cc graduated flask, dissolve in water and make up to the mark. Determine the specific gravity of the diluted solution by means of a pycnometer or Westphal balance. Ascertain from Table VI the percentage by weight of solids corresponding to the specific gravity of the diluted solution and calculate the total solids in the original sample by the formula: Solids in original sample = 4DS, D being the specific gravity of the diluted solution and S the per cent of solids in the diluted solution.

(b) CONFECTIONERY.

(1) *Lozenges and other pulverizable products.*

Weigh from 2 to 5 grams of the powdered sample in a tared platinum dish and dry to constant weight at 70° C. in vacuo or in the McGill oven.

(2) *Semiplastic, sirupy, or pasty products.*

Measure 25 cc of the 20 per cent solution, or mixture—2 (c) (1) (b)—into a tared platinum dish containing asbestos fiber and proceed as in 3 (a) (1).

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\*U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 28.

## 4.—DETERMINATION OF ASH.

Weigh out from 5 to 10 grams of molasses, honey, or sirup, or weigh 5 grams of the pulverizable confectionery, or measure 50 cc of the 20 per cent solution—2 (c) (1) (b)—into a tared platinum dish; evaporate to dryness on the water bath and burn slowly and cautiously over a low flame. After frothing has ceased, increase the flame and ignite to a white ash at a low, red heat.

In igniting saccharine products, frothing may be largely held in check by directing the flame at first down upon the mass from above instead of from under the dish, as ordinarily, until the material is well charred.

## 5.—EXAMINATION FOR MINERAL ADULTERANTS.

## (a) REDUCING TO ASH.

Comparatively large quantities of saccharine products may be readily and quickly reduced to an ash for mineral examination without the troublesome frothing that ordinarily ensues in igniting at once with a free flame by proceeding as follows:<sup>a</sup>

Mix 100 grams of molasses, sirup, or honey, or of the confectionery solution (2 (c) (1) (b)) evaporated to sirupy consistency, with about 35 grams of concentrated sulphuric acid in a large porcelain evaporating dish. An electric current is then passed through it while stirring, by placing one platinum electrode in the bottom of the dish near one side and attaching the other to the lower end of the glass rod, with which the contents are stirred. Begin with a current of about 1 ampere and gradually increase to 4.<sup>b</sup> In from ten to fifteen minutes the mass is reduced to a fine, dry char, which may then be readily burnt to a white ash in the original dish over a free flame or in a muffle.

If an electric current is unavailable, treat in a large porcelain evaporating dish 100 grams of the saccharine solution to be ashed, which should be evaporated to sirupy consistency if not already such, with sufficient concentrated sulphuric acid to thoroughly carbonize the mass, after which ignite in the usual manner.

Among the suspected adulterants to be looked for in the ash are salts of tin, used in molasses to bleach or lighten the color, and mineral pigments such as chromate of lead in yellow confectionery, and oxide of iron, the latter being commonly used as an intensifier of or substitute for the natural color of chocolate.

(b) DETERMINATION OF TIN IN MOLASSES<sup>c</sup> AND OTHER SACCHARINE PRODUCTS.<sup>d</sup>

Fuse the ash from a weighed portion of the sample with sodium hydroxid in a silver crucible, dissolve in water and acidulate with hydrochloric acid;<sup>e</sup> filter and precipitate the tin from this solution with hydrogen sulphid; wash the precipitate on a filter and dissolve it in an excess of ammonium sulphid. Filter this solution into a tared platinum dish and deposit the tin directly in the dish by electrolysis, using a current of 0.05 ampere. This current may be readily reduced from an ordinary 110-volt street circuit by means of a series of lamps, or a rheostat may readily be improvised for this purpose, consisting of a long, vertical glass tube, sealed at the bottom, containing a column of dilute acid through which the current passes, the

<sup>a</sup> Leach. 32d An. Rept. Mass. State Board of Health. (1900.) p. 653. Reprint, p. 37.

This method is preferred to the ordinary method of heating with sulphuric acid, especially in case of molasses, because, if properly manipulated, it so quietly comes into the form of a very finely divided char or powder, especially adapted for subsequent quick ignition.

<sup>b</sup> Modified from method of Budde & Schou for determining nitrogen electrolytically. *Ztschr. anal. Chem.*, 1899, **38**, 345.

<sup>c</sup> Leach. 31st An. Rept. Mass. State Board of Health, 1899, p. 625; Hilger & Laband, *Ztschr. für Untersuchung der Nahr.- u. Genuss.*, 1899, **2**, 795.

<sup>d</sup> This method is applicable also to condensed milk, canned goods, etc.

<sup>e</sup> See Methods for the examination of canned vegetables, p. 52.

resistance being changed by varying the length of the acid column contained between two electrodes immersed therein, one of which is movable. <sup>a</sup>

#### 6.—DETERMINATION OF ETHER EXTRACT IN CONFECTIONERY.

Measure 25 cc of the 20 per cent mixture or solution—2(c)(1)(b)—into a very thin, readily frangible glass evaporating shell (*Hoffmeister's Schälchen*), containing 5 to 7 grams of freshly ignited asbestos fiber; or, if impossible to thus obtain a uniform sample, weigh out 5 grams of the mixed, finely divided sample into a dish, and wash with water into the asbestos in the evaporating shell, using, if necessary, a small portion of the asbestos fiber on a stirring rod to transfer the last traces of the sample from dish to shell. Dry to constant weight at 100°, after which cool, wrap loosely in smooth paper, and crush into rather small fragments between the fingers, carefully transferring the pieces with the aid of a camel's hair brush to an extraction tube or a Schleicher and Schull cartridge for fat extraction. Extract with anhydrous ether or with petroleum ether in a continuous extraction apparatus for at least 25 hours. Transfer the solution to a tared flask, evaporate off the ether, dry in an oven at 100° C. to constant weight, and weigh.

Unless the ether is absolutely anhydrous, sugar will be dissolved. Ether which gives off hydrogen when treated with metallic sodium is unfit to use without purification. To purify it, let it stand for some time with calcium chlorid in the container, then pour off and distill over metallic sodium.

If petroleum ether is employed, it should be purified by fractional distillation so that it boils between 45° and 60° C. and leaves absolutely no residue.

#### 7.—DETERMINATION OF PARAFFIN IN CONFECTIONERY.

Add to the ether extract in the flask as above obtained, 10 cc of 95 per cent alcohol and 2 cc of 1:1 sodium hydroxid solution, connect the flask with a reflux condenser, and heat for an hour on the water bath or until saponification is complete. Remove the condenser, and allow the flask to remain on the bath till the alcohol is evaporated off and a dry residue is left. Treat the residue with about 40 cc of water and heat on the bath, with frequent shaking, till everything soluble is in solution. Wash into a separatory funnel, cool, and extract with four successive portions of petroleum ether, which are collected in a tared flask or capsule. Remove the petroleum ether by evaporation and dry in the oven to constant weight.

It should be noted that any phytosterol or cholesterol present in the fat would come down with the paraffin, but the amount would be so insignificant that except in the most exacting work it may be disregarded. The character of the final residue should, however, be confirmed by determining its melting point and specific gravity, and by subjecting it to examination in the butyro-refractometer. The melting point of paraffin is about 54.5° C.; its specific gravity at 15.5° is from 0.868 to 0.915, and on the refractometer (Zeiss's scale) the reading at 65° C. is from 11 to 14.5.

#### 8.—DETERMINATION OF NITROGEN.

Use 5 grams of the sample for this determination, and follow the details of the regular Gunning method. <sup>b</sup>

#### 9.—DETERMINATION OF STARCH IN CONFECTIONERY. <sup>c</sup>

Measure gradually 25 cc of the 20 per cent solution or uniform mixture (2 (c) (1) (b)) into a hardened filter or Gooch crucible, or transfer by washing 5 grams of the finely powdered substance to the filter or Gooch, and allow the residue on the filter to become air-dried. Extract with 5 successive portions of 10 cc of ether, then wash

<sup>a</sup> Wiley, Principles of Agricultural Analyses, vol. 3, p. 152.

<sup>b</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 16.

<sup>c</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 25.

with 150 cc of 10 per cent alcohol, and finally with 20 cc of strong alcohol. Transfer the residue to a large flask and boil gently for 4 hours with 200 cc of water and 20 cc hydrochloric acid (specific gravity 1.125), the flask being provided with a reflux condenser. Cool, neutralize with sodium hydroxid, add 5 cc of alumina cream, and make up the volume to 250 cc with water. Filter, and determine the dextrose in an aliquot part of the filtrate by Allihn's method, as directed in 13, page 49. The weight of the dextrose multiplied by 0.9 gives the weight of the starch.

#### 10.—POLARIZATION.

##### (a) MOLASSES.

Dissolve the normal weight of the sample (26.048 grams for the Schmidt and Haensch polariscope) in water in a 100-cc graduated flask, add an excess of lead subacetate solution,<sup>a</sup> and fill to the mark; shake to insure uniform solution, filter and polarize in a 100-mm tube, multiplying the reading by 2 for the direct polarization.

To 50 cc of the filtrate add 5 cc of concentrated hydrochloric acid. Heat slowly to 68° and cool. Polarize in the same tube at the same temperature as before, add 10 per cent to the reading, and multiply by 2 for the invert polarization.

The short tube (100-mm) is preferred for polarizing molasses not only on account of the more or less deep color of the clarified solution, but also because a molasses sample containing considerable commercial glucose would not read within the scale limits if the 200-mm tube were employed.

It sometimes happens, especially with molasses containing much glucose sirup, that it is impossible to obtain a clear filtrate after clarification with lead subacetate, or that the filtrate, at first clear, clouds up too quickly to admit of a satisfactory reading. In such cases weigh out a fresh portion of the sample, dilute, and add first the lead subacetate solution and then enough sodium sulphate or common salt to precipitate the excess of lead. Afterwards fill to the mark and proceed in the regular manner.

For medium or light-colored grades of molasses which yield but a small precipitate with lead subacetate, the above method of simple polarization both direct and invert gives results sufficiently accurate for ordinary work. For dark-colored or "black strap" molasses, or wherever extreme accuracy is required, employ the double-dilution method—10 (c).

##### (b) HONEY, MAPLE SIRUP, AND WATER-SOLUBLE CONFECTIONERY.

Follow directions given under "molasses," 10 (a), except that alumina cream<sup>b</sup> is employed in excess as a clarifier instead of subacetate of lead.

##### (c) CONFECTIONERY CONTAINING STARCH OR INSOLUBLE MATTER.

Employ the double-dilution method,<sup>c</sup> thus making due allowance for the volume of the precipitate. Take half the normal weight of the sample and make up the solution to 100 cc, using the appropriate clarifier (subacetate of lead for dark-colored confectionery or molasses, and alumina cream for light-colored confectionery and honey). Take the normal weight of the sample and make up a second solution with the clarifier to 100 cc. Filter and obtain direct polariscopic readings of both solutions. Invert each in the usual manner and obtain the invert readings of the two.

The true direct polarization of the sample is the product of the two direct readings divided by their difference. The true invert polarization is the product of the two invert readings divided by their difference.

<sup>a</sup> See footnote, page 84.

<sup>b</sup> Prepare by dividing a cold saturated aqueous solution of alum into two unequal portions, to the larger of which add a slight excess of ammonium hydroxid. Then add by degrees the remaining portion to a faint acid reaction. U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 39.

<sup>c</sup> Wiley & Ewell, Analyst, 1896, 21, 184.

## 11.—DETERMINATION OF CANE SUGAR.

Use Clerget's formula:

$$S = \frac{(a-b) 100}{144 - \frac{t}{2}}$$

where  $S$ =per cent of cane sugar,  $a$ =direct polarization,  $b$ =invert polarization,  $t$ =temperature.

12.—DETERMINATION OF COMMERCIAL GLUCOSE IN MOLASSES, SIRUPS, AND HONEY.<sup>a</sup>

As to preliminary indications of the presence of commercial glucose in these products, a sample of molasses of light color whose normal weight made up to 100 cc and polarized in a 200-mm tube shows a reading much in excess of 60° on the cane-sugar scale is almost sure to contain commercial glucose, while a dark-colored sample of molasses should, if pure, polarize considerably below 50° C. A sample of maple sirup which polarizes much in excess of 65° C on the cane-sugar scale is to be suspected of containing commercial glucose, while a sample of honey that polarizes to the right of the zero point is apt to be adulterated either with cane sugar or commercial glucose or both. If any of these products show an invert reading much to the right of the zero point, commercial glucose is almost sure to be present.

It is manifestly impossible to determine with absolute accuracy the amount of commercial glucose present by reason of the varying amount of dextrine, maltose, and dextrose present in the adulterant. It is possible, however, in molasses and maple sirup, wherein the amount of invert sugar is so small as not to appreciably affect the result, to estimate approximately the amount of commercial glucose by the following formula:

$$G = \frac{(a-S) 100}{175}$$

where  $G$ =per cent of commercial glucose,  $a$ =direct polarization,  $S$ =per cent of cane sugar.

In honey, which is composed largely of invert sugar, much closer results are attained by first inverting the sample and obtaining the polariscopic reading at 87° in a tube surrounded by hot water. This reading divided by 175 gives the approximate percentage of commercial glucose in the sample.

A large number of samples of commercial glucose have been procured by the department of food and drug inspection of the Massachusetts Board of Health directly from various manufacturers of compound or adulterated honey, molasses, and sirup, to ascertain the grade used by them for this purpose. As a result of this investigation, it has been found that the grade best adapted by its consistency for admixture with these products, and, indeed, the grade largely, if not universally, used for this purpose, has a density of about 42° Beaumé and polarizes on the cane-sugar scale at or about 175° (26.048 grams made up to 100 cc and polarized in a 200-mm tube with the Schmidt & Haensch instrument). From repeated experiments made in the writer's laboratory on mixtures containing known proportions of commercial glucose, 175 has been adopted as the most satisfactory factor and has been found to give a very close approximation.<sup>b</sup>

<sup>a</sup> Leach, 32d An. Rept. Mass. State Board of Health, 1900, p. 658. Reprint, p. 42.

<sup>b</sup> Among the samples of commercial glucose examined were several obtained from manufacturers of compound jellies and jams, and the examination of these would seem to show that the grade used mostly for this purpose polarizes at or about 150° C. If this is verified, 150 instead of 175 should be used in the above formula when applied to jellies and jams. The effect of high temperatures employed in the preparation of this class of goods should not be lost sight of, a factor that does not enter in to disturb the application of the method to adulterated molasses, sirups, and honeys which are mixed in the cold.

For chewing gum a grade of commercial glucose is used polarizing at about 185°.



For confectionery, as might be expected, from the wide variation in the character and consistency of candies, there is little uniformity in the grade of commercial glucose employed, so that it is not possible as in the case of molasses and honey to calculate the amount present. Nor is it so essential, in view of the fact that commercial glucose is rarely regarded as an adulterant of confectionery.

### 13.—DETERMINATION OF REDUCING SUGARS (ESTIMATED AS DEXTROSE).

Treat 5 grams of molasses, sirup, or honey, or 25 cc of the 20 per cent solution or mixture (2 (c) (1) (b)) or 5 grams of the powdered confectionery, with water in a 100-cc graduated flask, using 2 to 5 cc, of lead subacetate solution in the case of molasses or sirup, and 5 cc of alumina cream in the case of honey or confectionery. Make up to 100 cc, filter, take an aliquot part of the filtrate (25 to 50 cc), and make this up to 100 cc, the amount taken being such that when diluted the solution will contain not more than one per cent of dextrose. If lead subacetate has been used to clarify, add to the aliquot part taken, and before dilution, enough sodium sulphate to precipitate the excess of lead, then filter, and make up to the 100-cc mark.

Add 30 cc of Fehling's copper solution<sup>a</sup> to 30 cc of Fehling's alkaline tartarate solution<sup>b</sup> in a 250-cc Erlenmeyer flask.<sup>c</sup> Add 25 cc of the sugar solution (which must not contain more than 1 per cent of reducing sugar) with a burette, heat to boiling and boil exactly 2 minutes. Separate the precipitate as quickly as possible by filtering, with the aid of vacuum, through a layer of asbestos about 1 cm thick in a Gooch crucible (which with the asbestos has previously been ignited, cooled, and weighed), washing the cuprous oxide precipitate with boiling distilled water till the wash water ceases to be alkaline.

To prepare the asbestos, first boil it with nitric acid (sp. gr. 1.05 to 1.10), washing out the acid with hot water, then boil with a 25 per cent solution of sodium hydroxid and finally wash out the alkali with hot water. Keep the asbestos in water in a wide-mouthed flask or bottle, and transfer it to the Gooch by shaking it up in the water and pouring it quickly into the crucible while under suction.

Dry the Gooch with its contents in the oven, and finally heat it at dull redness for fifteen minutes. Transfer to the desiccator, cool, and weigh quickly as cupric oxid. A platinum Gooch may safely be used. If a porcelain Gooch is employed, extra precautions are necessary in heating to avoid cracking. With porcelain use a muffle.

Or, wash with alcohol and ether, dry for 20 minutes at 100° C. and weigh as cuprous oxid. In either case ascertain the weight of reducing sugar, in terms of dextrose, from Table VIII.

Or, the copper may be determined from the cuprous oxid in accordance with the official methods.<sup>d</sup>

### 14.—DETERMINATION OF ALCOHOL IN SIRUPS USED IN CONFECTIONERY ("BRANDY DROPS").<sup>e</sup>

Open each drop by cutting off a section with a sharp knife and collect in a beaker the sirup of from 15 to 25 of the drops, which will usually yield from 30 to 50 grams of sirup. Strain the sirup into a tared beaker through a perforated porcelain filter

<sup>a</sup> 34.639 grams  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , dissolved in water and diluted to 500 cc.

<sup>b</sup> 173 grams of Rochelle salts and 125 grams of potassium hydroxid dissolved in water and diluted to 500 cc.

<sup>c</sup> Defren, Jour. Am. Chem. Soc., 1896, 18, 749.

<sup>d</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46, p. 37.

<sup>e</sup> Thirty-second An. Rep. Mass. Board of Health, 1900, p. 757. Reprint, p. 41.

plate in a funnel to separate from particles of the inclosing shell, and ascertain the weight of the sirup. Dilute with half its volume of water and determine alcohol as directed on page 82.

#### 15.—DETECTION OF COLORING MATTER.

Proceed as directed under Coloring matters (p. 111 and following).

### VII. CANNED VEGETABLES.

By L. S. MUNSON,

*Bureau of Chemistry, U. S. Department of Agriculture.*

#### 1.—GENERAL DISCUSSION.

In the investigation of canned vegetables, the proximate analysis is, as a rule, of little value in determining quality; much more depends upon the size, age, and fresh and healthy condition of the vegetables at the time of canning, and the treatment during and subsequent to the processes of canning. Hence, methods for the proximate analysis have been given minor consideration to means of determining the quality of the various classes of vegetables and the detection of different forms of adulterants. Much still remains to be done with this class of food, as the time at the disposal of the writer was not sufficient to investigate thoroughly the various problems that presented themselves.

#### 2.—MACROSCOPIC EXAMINATION.

A careful macroscopic examination is often of material value in detecting inferior quality with certain classes of vegetables. Upon opening a can, carefully note the appearance of the contents as to quality, color, and size. Any undue corrosion, or blackening of the walls of the can, should also be observed. With mushrooms and capers, no further examination is necessary, as a rule, except the detection of sulphites in the former.

The most common form of mushrooms found upon the market is *Agaricus campestris*, although different varieties of *Boletus* are occasionally found. The latter are particularly susceptible to attack by larvæ and, except in a fresh state, are seldom free from them. These larvæ may readily be seen with the naked eye, or by use of a small hand lens. Many of the mushrooms on the market are of inferior quality, and consist largely of old and broken fragments of tops and stems; occasionally diseased fungi are to be found in the inferior grades. Owing to the nature of this vegetable, only the fresh, healthy specimens should be passed as edible.

Capers are the flower buds of *Cappares spinosa* and, so far as known, are but little liable to adulteration. Owing to their green color, it is always advisable to make a qualitative test for copper.

Olives are to be judged entirely by general appearance and by taste. Gherkins and mixed pickles, while not strictly under this class of foods, are considered here along with olives for the sake of completeness; these also are to be judged largely macroscopically and by taste. The use of copper with this class is of frequent occurrence to produce the bright green color; with mixed pickles, where mustard is used, turmeric is frequently added as a coloring agent. It is also advisable to test for aniline dyes where turmeric is not detected.

#### 3.—PREPARATION OF THE SAMPLE.<sup>a</sup>

Weigh the full can; open, pour off the liquid portion, and reweigh the can; then empty out the solid contents of the can and weigh again. From these weights estimate the percentage of liquid and solid contents. By this means, any undue propor-

<sup>a</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 8, p. 1027.

tion of liquor, owing to excess of water added, will be detected. Then thoroughly grind the entire contents of the can, either in a mortar or by means of a food chopper; mix thoroughly and preserve in a glass-stoppered bottle for analysis. Unless the analysis is to be completed within a reasonably short time it is best to dry the entire sample after the determination of moisture is made. After thorough drying, the material is allowed to stand exposed to the air for several hours, or until it has become air dry. A second moisture determination is necessary with this procedure.

#### 4.—PROXIMATE ANALYSIS.

For methods of proximate analysis, see Bulletin 13, part 8, U. S. Department of Agriculture, Division of Chemistry, page 1028.

#### 5.—DETECTION OF SACCHARIN.

Saccharin is quite extensively used in canned sweet corn as a sweetening agent. For its detection add from 25 to 40 cc of water to about 20 grams of the sample; macerate and strain through muslin; acidify with 2 cc of sulphuric acid (1 to 3) and extract with ether. Separate the ether layer, allow the ether to evaporate spontaneously, and take up the residue with water. If saccharin be present its presence will be indicated by the sweet taste imparted to the water. To confirm this test add from 1 to 2 grams of sodium hydroxid, and place the dish in an oil bath. Maintain the temperature of the oil at 250° C. for twenty minutes, when the saccharin will be converted into salicylic acid. After cooling and acidifying with sulphuric acid extract in the usual way and test for salicylic acid. This test, of course, presupposes the absence of salicylic acid in the original sample. If salicylic acid is present in the original sample it must be removed before making the test for saccharin.

#### 6.—DETERMINATION OF SULPHITES.

Sulphites are largely used with certain classes of vegetables, notably corn and asparagus, as a bleaching agent; they may also find use with this class of foods as a preservative.

##### (a) DISTILLATION METHOD.

For their determination place 50 grams of the material in a distilling flask, add about 5 cc of a saturated solution of glacial phosphoric acid, and proceed with the distillation and the titration of the sulphite as directed on p. 90. Where only a qualitative test is desired, take the first few cubic centimeters of the distillate, add a slight excess of iodine solution, boil to expel the excess of iodine, then acidify with hydrochloric acid and add barium chlorid solution. This test is very delicate and is easily applied.

##### (b) REDUCTION METHOD.\*

To about 25 grams of the sample placed in a 200-cc Erlenmeyer flask add some pure zinc and several cubic centimeters of hydrochloric acid. In the presence of sulphites, hydrogen sulphid will be generated and may be tested for with lead paper. Traces of metallic sulphids are occasionally present in vegetables, and by the above test will indicate sulphites. Hence positive results obtained by this method should be verified by the distillation method.

It is always advisable to make the quantitative determination of sulphites, owing to the danger that the test may be due to traces of sulphids. A trace is not to be considered sufficient either as a bleaching agent or as a preservative.

#### 7.—DETECTION OF PRESERVATIVES.

See methods given under Preservatives (p. 107, and following).

\*Dept. of Agr., Div. of Chem., Bul. 13, pt. 8, p. 1032.

## 8.—DETECTION OF COLORING MATTERS.

(a) IN TOMATOES<sup>a</sup> AND CATSUPS.

Most of the coloring matter used in tomatoes is either of coal-tar origin or cochineal, and the general methods given under Coloring Matter (p. —) may be applied in these cases. Extract the color from the dried pulp with ordinary alcohol after acidifying with hydrochloric acid and filter. Eosin gives a characteristic fluorescent filtrate. Dilute the filtrate with water, extract with amyl alcohol and dye. Cochineal if present is in the form of a lake and will require strong hydrochloric acid to decompose it. After extraction with amyl alcohol it may be tested with uranium acetate (p. 120).

## (b) IN PEAS, BEANS, GHERKINS, ETC.

Copper salts are most commonly employed in this class of goods, although it is said that zinc is occasionally used. For the qualitative detection, ash from 15 to 20 grams of the sample, either with or without previous treatment with concentrated sulphuric acid (see Heavy Metals below), transfer the ash to a beaker and treat with nitric acid; filter, make the filtrate alkaline with ammonia, and if a precipitate forms filter again. Copper will be indicated by the blue color of the filtrate. If further test is desired acidify with acetic acid, and add potassium ferro-cyanide. Red coloration or precipitate verifies the test.

## (c) IN MIXED PICKLES, ETC.

Turmeric is frequently used and may be identified by the method given under Coloring Matter (p. 120).

## 9.—DETERMINATION OF TOTAL AND VOLATILE ACIDITY.

It is occasionally desirable to determine total acidity in tomatoes and catsups, and volatile acidity in the latter. For this purpose use methods described under Fermented and Distilled Liquors (p. 83). Express fixed acids as citric; one 1 cc of decinormal alkali equals .0070 gram of citric acid. Express volatile acids as acetic; 1 cc of decinormal alkali equals .0060 gram of acetic acid.

## 10.—DETERMINATION OF HEAVY METALS.

Owing to the almost universal presence of tin, the frequent occurrence of lead and zinc, and the extensive use of copper as a coloring agent in this class of food materials, the determination of heavy metals is of particular value. The method described by Allen<sup>b</sup> and modified by Bigelow and Munson, has been used in the laboratory of the Bureau of Chemistry for the determination of heavy metals in canned meats, and may be applied as well to vegetables. Since the work on canned meats, however, the writer has worked out a method that for accuracy and ease of manipulation is preferred to the modified Allen's method.

(a) ALLEN'S METHOD, MODIFIED BY BIGELOW AND MUNSON.<sup>c</sup>

Treat 100 grams of the moist material, or 25 grams of the dried material, with about 5 cc of concentrated sulphuric acid and 2 cc of nitric acid. After foaming has ceased add 3 grams of magnesium oxid and mix thoroughly. Then ignite over a Bunsen burner or, preferably, in a muffled furnace, until thoroughly charred. Grind in a mortar, and again ignite to complete combustion. The addition of a few drops of nitric acid may be necessary toward the end to complete the operation. Add

<sup>a</sup> Girard and Dupré. *Analyses des matières alimentaires*, etc.

<sup>b</sup> Allen's *Com. Organic Anal.* 3d ed. Vol. IV, p. 299.

<sup>c</sup> *Jour. Amer. Chem. Soc. Proc.* 1900, 22, 32.

about 50 cc of hydrochloric acid (1:3) and heat to boiling or upon a steam bath for a half hour. Nearly neutralize the acid with sodium hydroxid dilute to 150 cc with water, precipitate with hydrogen sulphid, and filter, after heating for a few moments upon a steam bath to facilitate the separation of the precipitated sulphids. Dry the precipitate and insoluble ash residue, and then fuse in a porcelain crucible with a mixture consisting of one gram each of sodium carbonate, potassium carbonate, and sulphur. Dissolve the fused mass with hot water and filter. Sulphids of lead and copper remain upon the filter. Acidify the filtrate with acetic acid to precipitate the tin sulphid. Collect the tin sulphid upon a filter. Wash thoroughly, and then dissolve by the aid of heat in a concentrated solution of ferric chlorid. The reduced iron salt is then titrated with potassium dichromate.\* One cc of decinormal potassium dichromate equals 0.00295 grams of tin. The determination of the tin by igniting and weighing as stannic oxid was found to be unreliable, owing to the precipitation of appreciable amounts of silica that was dissolved by the mixed carbonates from the porcelain crucible. Determine the copper and lead, which remain as insoluble sulphids after the fusion, and the zinc, which remains in the original filtrate, according to the scheme described under the following method.

(b) MUNSON'S METHOD.

Treat 100 grams of the moist sample after evaporation to dryness, or 25 grams of the dry sample in a four-inch porcelain evaporating dish with sufficient concentrated sulphuric acid to thoroughly carbonize the mass. Usually from 10 to 15 cc are sufficient for this purpose. Gently heat over a Bunsen burner until all danger of foaming is past, which will require not more than three minutes; then transfer the dish to a muffle furnace and keep it at a low red heat until all organic matter is destroyed. It is occasionally found necessary to add a few drops of nitric acid to completely destroy organic matter. When the material is completely ashed, allow the dish to cool; add 25 cc of hydrochloric acid (1 to 8) and evaporate on a water bath to dryness; take up with water and acidify with two or three drops of hydrochloric acid. Transfer to a beaker without filtering and treat with hydrogen sulphid. After heating upon a water bath for a few minutes the precipitate and the insoluble residue are collected upon a filter. The precipitate and residue may contain sulphids of tin, lead, and copper, and oxid of tin; the filtrate will contain any zinc that is present. Fuse the sulphid precipitate and insoluble ash residue with about three grams of caustic soda in a silver crucible for a half hour to render soluble any insoluble tin compounds. Dissolve the mass with hot water and slightly acidify with hydrochloric acid. Again treat with hydrogen sulphid without filtering. By this treatment all the tin is thrown down as sulphid with the sulphids of copper and lead. Collect the precipitate upon a filter and wash thoroughly with hot water. The filtrate may be rejected. To separate the tin sulphid from those of copper and lead, wash several times upon the filter with separate portions of 10 cc of strong boiling ammonium sulphid. Usually 50 cc of the ammonium sulphid will be found sufficient to completely dissolve all tin sulphid; but portions of the filtrate should be tested to make sure of this point. The filtrate is then made acid with hydrochloric acid to precipitate the tin sulphid, which, after standing for a few moments, is collected upon an ashless filter, ignited, and weighed as stannic oxid.

Treat the insoluble residue remaining from the ammonium sulphid washing with nitric acid, filter, wash, nearly neutralize with ammonia the excess of mineral acid, and add ammonium acetate, as there is usually a small amount of iron present. If any iron salt precipitates, filter, wash and divide the filtrate for the determinations of copper and lead. In the absence of lead, copper may be determined electrolytically, or it may be titrated with potassium cyanid. Unless added as a coloring agent, copper will seldom be present in sufficient quantity to warrant its determination.

\* Sutton, Volumetric Analysis, 8th ed., p. 373.

Precipitate lead with potassium chromate in an acetic acid solution; and weigh upon a tared filter as lead chromate.

Evaporate the filtrate from the hydrogen sulphid precipitate to about 60 cc; add bromin water to oxidize the iron salts, and any remaining hydrogen sulphid. Boil off the excess of bromin and, unless the solution is distinctly yellow, add a few drops of concentrated solution of ferric chlorid to make it so. Nearly neutralize the mineral acid with ammonia, and add ammonium acetate to precipitate iron phosphate and excess of iron. Filter and thoroughly wash the precipitate. To the filtrate, made distinctly acid with acetic acid and boiled, add hydrogen sulphid to precipitate zinc. Unless the zinc sulphid comes down white, it should be dissolved, again treated with ammonium acetate to remove traces of iron, and re-precipitated as sulphid. Finally collect the zinc sulphid upon an ashless filter, ignite and weigh as zinc oxid.

#### 11.—“SOAKED” VEGETABLES.

A class of canned vegetables commercially known as “soaked” goods is now very commonly found upon the market, and constitutes the cheapest grade of vegetables sold. So far as the writer's experience goes, only peas, beans, and corn, or combinations of these three, are found in this class. The material used for “soaked” products are the ordinary matured peas and beans, such as are used for seed, or are sold dried upon the market, and corn that has passed the stage when it can be supplied for the green market. The particular advantage in canning these goods is that the season for green vegetables passes rapidly, and in case the supply is greater than the canneries can handle, recourse is made to the packing of the matured product. Besides, these dried materials may be kept for some time, and thus serve to keep the canneries in operation during the less busy season.

So far as the composition of this class of canned vegetables is concerned, it probably varies but little from that of the younger vegetables, yet it does not possess the value as a relish that the former has. In the mature vegetables the percentage of total solids is much higher than in the young and more succulent vegetables, and this condition holds in the canned goods if only the solid contents of the can are considered. However, in a large number of samples of “soaked” goods examined, the proportion of liquid to solid portion was exceedingly high; so that when the entire contents of the can were taken the per cent of total solids was about normal for the green vegetables.

The detection of “soaked” vegetables is not a difficult matter for one who has had experience with this class of goods, but for a layman the task may not be so easy. As stated above, the high percentage of solids in the solid portion of the can is characteristic. Soaked peas and beans lose much, if not all, of their green color, and have the general appearance of the well-matured product. Their cotyledons are well formed, firm and mealy. With the pea the caulicle is particularly prominent, the process of soaking having been sufficient to start its development. With corn, the kernel is plump and hard and lacking in milky consistency. The succulence so characteristic of the green pea, bean, and corn is entirely lacking. The sense of taste may also be applied in the detection of this class of goods. From their nature it is difficult to apply specific tests, but a little practice will enable the analyst to detect them with reasonable certainty.

### VIII.—COCOA AND ITS PREPARATIONS.

By F. T. HARRISON,

*District Analyst, London, Ontario.*

It has been found impossible to prepare the report on this subject this year. The heading is inserted here to preserve its proper order.

**IX.—TEA AND COFFEE.**

By W. H. ELLIS,  
*District Analyst, Toronto, Canada.*

It has been found impossible to prepare the report on this subject this year. The heading is inserted here to preserve its proper order.

**X. SPICES.**

By A. L. WINTON,  
*Chemist of State Experiment Station, New Haven, Conn.*

**1.—GENERAL DISCUSSION.**

The microscope is a most valuable means of detecting adulterants of vegetable origin in spices, as it usually discloses the particular adulterant present, even when in small amount.

Quantitative determinations are made, either to corroborate the results of the microscopical examination, or to detect exhausted spices, mineral matter, and other adulterants which do not have distinctive microscopic characters.<sup>a</sup>

**2.—PREPARATION OF SAMPLES.**

Grind the sample so as to pass a sieve with round holes one millimeter in diameter. For the determination of starch in pepper by the diastase method, reduce a portion of the sample to an impalpable powder, by grinding in a mortar.

**3.—DETERMINATION OF WATER.<sup>a</sup>**

Dry two grams at 110° C. to constant weight. From the loss in weight thus sustained subtract the amount of volatile ether extract determined as below described. This method, described by Richardson,<sup>b</sup> gives a close approximation to the true percentage of moisture.

**4.—DETERMINATION OF TOTAL ASH.**

Follow the method of the Association.<sup>c</sup> If calcium carbonate is present, care must be taken to burn the material and also the residue after exhaustion with water, at a heat below redness, thus avoiding loss of carbonic acid of the carbonate. When leaching with water is necessary, it is advisable to add a few drops of ammonium carbonate solution before evaporation.

**5.—DETERMINATION OF ASH SOLUBLE IN WATER.**

Boil the ash prepared as above with 50 cc of water, collect the insoluble portion in a Gooch crucible, wash with hot water, dry, ignite, and weigh.<sup>d</sup> Subtract the percentage of insoluble ash thus determined from the percentage of total ash, thus obtaining the percentage of water-soluble ash.

**6.—DETERMINATION OF "SAND" OR ASH INSOLUBLE IN HYDROCHLORIC ACID.**

Incinerate 2 grams of the material as above directed, boil with 25 cc of 10 per cent hydrochloric acid (sp. gr. 1.050) for 5 minutes, collect the insoluble matter in a Gooch crucible, wash with hot water, ignite, and weigh.

<sup>a</sup> See also Appendix, p. 152.

<sup>b</sup> U. S. Dept. Agr., Div. Chem., Bul. 13, Part 2, p. 165.

<sup>c</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 23.

<sup>d</sup> Conn. Agr. Expt. Sta. Rept. 1898, p. 186.

## 7.—DETERMINATION OF LIME.

Calcium sulphate and carbonate are frequently used as adulterants, and are also present in appreciable amount in limed nutmegs, ginger, etc. In the presence of calcium sulphate, the water solution gives tests for both lime and sulphuric acid. Samples containing a considerable amount of carbonate effervesce on addition of 10 per cent hydrochloric acid. Determine lime in the ash, after separation of iron and alumina phosphates, as described under Baking Powder (p. 106.)

8.—DETERMINATION OF TOTAL SULPHUR.<sup>a</sup>

*(For mustard and samples adulterated with calcium sulphate.)*

Convert about 10 grams of sodium peroxid into hydroxid in a nickel crucible by adding a little water and boiling over a low flame until the excess of water is expelled. Stir 1 gram of the material into the slightly cooled hydroxid and oxidize by gradually raising the heat and adding small portions of sodium peroxid until the oxidation is complete. Dissolve the fused mass in 400 cc of water, acidify strongly with hydrochloric acid, boil until the excess of peroxid is destroyed and chlorine expelled, filter through pure paper, make neutral with ammonia, and add an excess of 4 cc of concentrated hydrochloric acid. From the boiling solution precipitate sulphuric acid by gradually adding a solution containing 1 gram of barium chlorid. After standing over night, in a warm place, filter the barium sulphate, wash, ignite, and weigh.

Osborne has found commercial sodium peroxid to be freer from sulphur than most preparations of so-called chemically pure sodium hydroxid made from the metal, and as the former is very much cheaper than the latter, it is advantageous to use it as here described.

## 9.—DETERMINATION OF CARBON DIOXID.

*(For samples adulterated with calcium carbonate.)*

Proceed as directed under total carbon dioxid in Baking Powder (p. 98 and following).

10.—DETERMINATION OF VOLATILE AND NONVOLATILE ETHER EXTRACT.<sup>b</sup>

Extract 2 grams of the ground material for 20 hours, in a continuous extraction apparatus, with absolute ether.<sup>c</sup> Transfer the ethereal solution to a tared capsule and allow to evaporate at room temperature. Let stand 18 hours over sulphuric acid and weigh the total ether extract. Heat the extract gradually to 100° C., continue the heating at that temperature for 6 hours, and then at 110°, until the weight becomes constant. The loss is volatile oil; the residue, nonvolatile ether extract.

11.—DETERMINATION OF ALCOHOL EXTRACT.<sup>d</sup>

Place 2 grams of material in a 100-cc flask and fill to the mark with 95 per cent alcohol by volume (sp. gr. 0.815 at 15.5° C.). Stopper, shake at intervals of 30 minutes for 8 hours, and allow to stand 16 additional hours without shaking. Filter the extract through a dry filter, evaporate 50 cc to dryness in a flat-bottomed dish on a water bath, and heat to constant weight at 110° C. The result is practically the same when the time of extraction is 48 instead of 24 hours. Winton, Ogden, and Mitchell,<sup>e</sup> who describe this method, do not claim that it extracts all matter soluble in alcohol; in fact, the residue separated from the solutions by filtration, when

<sup>a</sup> Osborne, Conn. Agr. Expt. Sta. Rept., 1900, p. 445.

<sup>b</sup> Richardson, U. S. Dept. of Agr., Div. of Chem., Bul. 13, Part 2, p. 165.

<sup>c</sup> See Appendix, pp. 153 and 154.

<sup>d</sup> See Appendix, p. 154.

<sup>e</sup> Conn. Agr. Expt. Sta. Rept., 1898, p. 187.



treated for 24 additional hours with a fresh portion of alcohol, yielded, in their experience, small additional amounts of extract. The method, however, gives nearly the full amount of extract and the results are concordant; whereas, extraction in a Soxhlet apparatus, if continued until no more extract is removed, is an interminable operation, and, as it is difficult to keep the strength and temperature of the extracting alcohol constant, gives results far from satisfactory.

## 12.—DETERMINATION OF COPPER-REDUCING MATTERS BY DIRECT INVERSION.<sup>a</sup>

Extract 4 grams of the material on a Schleicher and Schuell's No. 589 blue-ribbon washed filter, or some other filter that will completely retain the smallest starch granules, with five successive portions of 10 cc of ether. After the ether has evaporated, wash with 150 cc of 10 per cent (by volume) alcohol. Weak alcohol is employed instead of water, because, as pointed out by Lindsey, it is not so liable to carry starch granules through the paper.

Since it is not possible to wash samples of Batavia cassia with water or dilute alcohol, owing to the formation of a glutinous mass which clogs the filter, for the sake of uniformity, all preliminary washing is best omitted in determinations made on all varieties of cassia, as well as on cassia buds and cinnamon.

Carefully wash the residue from the paper into a 500-cc flask, with 200 cc of water, using a small wash bottle, and gently rubbing the paper with the tip of the finger.

Convert the starch into dextrose by the Sachsse method,<sup>b</sup> as follows:

Add 20 cc of 25 per cent hydrochloric acid (sp. gr. 1.125) and heat for three hours on a boiling water bath. Cool the solution nearly, but not quite, neutralize with sodium hydroxid solution, make up to 500 cc, and filter through a dry paper.

Determine reducing matters by the Allihn method,<sup>c</sup> as follows:

Mix 30 cc of a solution containing 173 grams of Rochelle salts and 125 grams of caustic potash in 500 cc of water, and 30 cc of a solution of 34.69 grams of pure crystallized copper sulphate in 500 cc of water, in a beaker of 200 cc capacity and heat to boiling. To the boiling liquid, without delay, add 25 cc of the solution to be examined, and continue the heating until boiling begins again. After the reduced copper suboxid has settled, collect on a Gooch crucible, dry at a moderate heat, and finally heat for three to five minutes at dull redness, taking care to avoid a bright red heat and to allow access of sufficient air to complete the oxidation to copper oxid (after Bartlett<sup>d</sup>). After weighing, repeat the heating to make certain that the oxidation is complete.

From the weight of copper oxid calculate the weight of metallic copper, using the factor 0.7986, and find the corresponding amount of dextrose in Table VIII. To obtain the corresponding weight of starch, multiply the weight of dextrose by 0.9.

If desired, the copper may be weighed as  $\text{Cu}_2\text{O}$  after washing with alcohol and drying at 100° C., or it may be determined electrolytically by one of the official methods.

To prepare asbestos pulp for use in the Gooch crucible, cut woolly asbestos (best quality) into small pieces, boil with hydrochloric acid, and wash free from acid and fine particles on a sieve with one-mm meshes. Woolly asbestos of suitable quality, when packed in the crucibles with the aid of a blunt glass rod, retains completely the finely divided copper suboxid, which is not true of the variety usually employed in filtering coarser precipitates.

Copper-reducing matters by direct inversion was first determined in pepper by

<sup>a</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 13: p. 166. Conn. Agr. Expt. Sta. Rept., 1898, p. 187. See also Appendix, p. 154.

<sup>b</sup> Chem. Centralbl. 1877, 8, 732.

<sup>c</sup> Jour. prakt. Chem., 1880, N. F., 22, 52.

<sup>d</sup> Maine Agr. Expt. Sta. Rept., 1888, p. 207.

Lenz.<sup>a</sup> Although useful, the results are not of as great value as those by the diastase method.

### 13.—DETERMINATION OF STARCH BY DIASTASE METHOD.<sup>b</sup>

Extract 4 grams of the finely pulverized material with ether and 10 per cent alcohol, as described in the preceding section. Carefully wash the wet residue from the paper into a beaker with 100 cc of water, heat on an asbestos plate to boiling with constant stirring, and continue the boiling and stirring thirty minutes. Replace the water lost by evaporation, and immerse the beaker in a water bath kept at from 55 to 60°. When the liquid has cooled to the temperature of the bath, add 10 cc of fresh extract of malt (prepared by digesting for two or three hours 100 grams of powdered fresh malt with 1,000 cc of water and filtering), and digest the mixture for one hour, with occasional stirring. Boil a second time for fifteen minutes, cool, and digest as before with another 10-cc portion of malt extract. Heat to boiling the third time, cool, and make up the liquid to 250 cc in a graduated flask, filter through a dry paper, and remove 200 cc of the filtrate to a 500-cc flask. Conduct the inversion with acid, and determine the reducing power of the solution, as already described under "Copper-reducing matters by direct inversion," making a correction for the copper reduced by the added malt extract, as determined by blank analyses. The residue after the malt digestion, when examined microscopically, must be entirely free from starch.

Results by Winton, Ogden, and Mitchell<sup>c</sup> show that cayenne pepper, mustard, and certain other materials, which are practically free from starch, yield very little or no copper-reducing matter, when treated by the method just described. This treatment is, therefore, without effect on the cellulose, pentosans, or other matters in the spices named, although they yield copper-reducing material on treatment with acid.

On the other hand, in decorticated white pepper and Jamaica ginger, which contain little besides starch that is affected by acid, practically the same results are obtained by the diastase method as by direct inversion with acids.

This determination of starch is very valuable as a means of detecting starchy adulterants in spices normally free from starch and nonstarchy adulterants in spices which contain starch.

### 14.—DETERMINATION OF CRUDE FIBER.<sup>d</sup>

The method is that adopted by the Association of Official Agricultural Chemists for the analysis of cattle foods, except that the fiber is filtered and weighed on a paper rather than on a Gooch crucible, since the latter is liable to clog, rendering filtration impossible. Place the residue from the determination of ether extract in a 500-cc Erlenmeyer flask, and add 200 cc of boiling 1.25 per cent sulphuric acid. Loosely cover the flask, heat at once to gentle boiling, and continue the boiling thirty minutes. Filter on a paper, wash with hot water, and rinse back into the same flask with 200 cc of boiling 1.25 per cent sodium hydroxid solution, nearly free from carbonate. After boiling, as before, for thirty minutes, collect the fiber on a weighed paper, thoroughly wash with hot water, and finally with a little alcohol and ether. Dry to constant weight at 100° C., and weigh. Deduct the amount of ash in the fiber, as determined by incineration, from the total weight.

Determine the loss in weight sustained by the paper on treatment with sodium-hydroxid solution, alcohol and ether, and introduce the necessary correction.

<sup>a</sup> Ztschr. anal. Chem., 1884, **23**, 501.

<sup>b</sup> Maercker, *Handbuch der Spiritusfabrikation*, 7th ed., 1898, p. 109; Wiley, *Principles and Practice of Agricultural Analysis*, 1898, Vol. III, p. 198.

<sup>c</sup> Conn. Agr. Expt. Sta. Rept., 1898, p. 189.

<sup>d</sup> See Appendix, pp. 154 and 155.

## 15.—DETERMINATION OF NITROGEN.

## (a) KJELDAHL METHOD.

(For all spices except black and white pepper.)

See methods of the Association of Official Agricultural Chemists.<sup>a</sup>

## (b) GUNNING-ARNOLD METHOD.

(For black and white pepper.)

Owing to the presence of piperine, the Gunning-Arnold method<sup>b</sup> must be used to determine nitrogen in both black and white pepper. Mix 1 gram of the material in a 600-cc Jena<sup>c</sup> flask with 1 gram of copper sulphate, 1 gram of mercuric oxid, 15 to 18 grams of potassium sulphate, and 25 cc of sulphuric acid. After heating gently until frothing ceases, boil the mixture from two to four hours. When nearly cool, add about 300 cc of water, 50 cc of potassium sulphid solution (40:1,000), and sodium hydroxid solution to alkaline reaction. Distill into standard acid and titrate with standard alkali, as usual.

## 16.—DETERMINATION OF NITROGEN IN NON-VOLATILE ETHER EXTRACT.—

WINTON, OGDEN, AND MITCHELL METHOD.

(For black and white pepper.)

Extract 10 grams of pepper for twenty hours in a continuous extraction apparatus with absolute ether, collecting the extract in a weighed Jena flask of about 250-cc capacity. Evaporate the ether, dry first at 100° C., and finally to constant weight at 110° C. Determine the nitrogen in the weighed extract by the Gunning-Arnold method, digesting in the same flask used for the extraction. Calculate the parts of nitrogen in 100 parts of the non-volatile ether extract.

Results on authenticated samples show that 100 parts of non-volatile ether extract from black pepper, owing to the presence of piperin, contain not less than 3.25 parts, and from white pepper not less than 4.00 parts of nitrogen. Linseed meal and other oily adulterants may contain about the same amount of ether extract as pepper, but this extract is practically free from nitrogen.

If desired, crude piperine may be calculated from the nitrogen by multiplying by 20.36.

## 17.—DETERMINATION OF COLD WATER EXTRACT.

(For ginger.)

Place 4 grams of ginger in a graduated 200-cc flask, add water to the mark, shake at half-hour intervals during 8 hours and let stand 16 additional hours, without shaking. Filter and evaporate 50 cc to dryness in a flat-bottomed metal dish. Dry to constant weight at 100° C.

Cold water extract was first determined by Allen and Moor<sup>d</sup> as a means of detecting exhausted ginger. The process of Winton, Ogden, and Mitchell<sup>e</sup> here described is more easily carried out and gives more concordant and somewhat higher results than extraction on a filter with the same volume of water added in consecutive portions. Complete extraction on a filter was found impracticable.

<sup>a</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 14.

<sup>b</sup> Ztschr. anal. Chem. 1892, **31**, 525; Conn. Agr. Expt. Sta. Rept., 1898, p. 190.

<sup>c</sup> See Appendix, p. 155.

<sup>d</sup> Analyst, 1894, **19**, 124.

<sup>e</sup> Conn. Agr. Expt. Sta., Rept. 1898, p. 190.

18.—DETERMINATION OF TANNIN EQUIVALENT BY THE LÖWENTHAL-RICHARDSON METHOD.<sup>a</sup>

(For cloves and allspice.)

Extract 2 grams of material 20 hours with absolute ether. Boil the residue 2 hours with 300 cc of water, cool, make up to 500 cc and filter. Measure 25 cc of this infusion into a flask of about 1,200-cc capacity, add 20 cc of indigo solution and 750 cc of distilled water. Run in standard permanganate solution at the rate of one or two drops a second, with constant shaking, until a bright golden yellow color appears.

Determine in the same manner the number of cubic centimeters of permanganate solution consumed by 20 cc of indigo solution alone and subtract from the number consumed by the spice infusion and indigo solution together.

Indigo solution must be made from sodium sulphindigotate of best quality (such as is furnished by Gehe & Co., Dresden, or Grueber & Co., Leipzig), otherwise the titration will not be sharp. Dissolve 6 grams of the salt in 500 cc of water, with the aid of heat. Cool, mix with 50 cc of concentrated sulphuric acid, make up to 1 liter and filter.

Prepare standard potassium permanganate solution by dissolving 1.333 grams of the pure salt in 1,000 cc of water. Standardize by titration of 10-cc portions of decinormal oxalic acid solution (6.3 grams of the pure crystallized acid in 1,000 cc), which have been previously diluted to 500 cc, heated to 60° C. and mixed with 20 cc of dilute sulphuric acid (1:3 by volume). Add the permanganate solution slowly, with constant stirring, until a pink color appears. Ten cubic centimeters of decinormal oxalic acid solution are equivalent to 0.06232 gram of quercitannic acid, or 0.008 gram of "oxygen absorbed."

Ellis first recommended the determination of tannin as a means of detecting adulteration, but Richardson found that the abbreviated method here described is quite as useful as the more tedious process for the actual determination of tannin.

## 19.—MICROSCOPICAL EXAMINATION.

As already stated, the microscope is the most valuable means of detecting adulterants of vegetable origin in spices, as it usually discloses the particular adulterant present, even when in small amount. The analyst who undertakes this work should have a general knowledge of vegetable histology and microscopic manipulation, and should be thoroughly familiar with the microscopic appearance of the spices and the spice adulterants. He should have at his command the standard works on these subjects, and also a set of standard samples of all the materials likely to be encountered. The following works are especially recommended:

*Moeller*. Mikroskopie der Nahrungs- und Genussmittel aus dem Pflanzenreiche. Berlin, 1886.

*Vogl*. Die wichtigsten vegetabilischen Nahrungs- und Genussmittel. Berlin, 1899.

*Tschirch und Oesterle*. Anatomischer Atlas der Pharmakognosie und Nahrungsmittelkunde. Leipzig, 1900.

Only a few general hints are here given:

## (a) APPARATUS.

Dissecting microscope or hand lens.

Compound microscope provided with  $\frac{2}{3}$  and  $\frac{1}{8}$ -inch objective, 1 and 2-inch oculars, double nosepiece, eyepiece micrometer, and polarizing apparatus.

A series of sieves with meshes ranging from 0.2 to 2 mm.

Slides, cover glasses, needles, scalpels, forceps, etc.

<sup>a</sup> Allen. Commercial Organic Analysis, 1889, Vol. III, Part I, p. 109. U. S. Dept. of Agr., Div. of Chem., Bul. 13, Part 2, p. 167.

## (b) MICRO-REAGENTS.

Of the numerous reagents employed in histological work, the following are most useful in spice examination:

Distilled water.

Glycerin, pure and diluted with equal volume of water.

Absolute alcohol.

Ether.

Ammonium hydroxid.

Potassium hydroxid (5 per cent).

Chloral hydrate (8 parts in 5 parts of water).

Schultze's macerating mixture: Crystallized potassium chlorate mixed with nitric acid as needed.

Iodin in potassium iodid: 0.05 gram iodine, 0.2 gram potassium iodid, and 15 cc water.

Chlorzinc iodine: Treat an excess of zinc with hydrochloric acid, evaporate to a thick sirup and filter through asbestos. As needed, saturate a small portion of the solution first with potassium iodid and finally with iodine.

Millon's reagent: Dissolve metallic mercury in its weight of concentrated nitric acid, add an equal volume of water, and decant off the clear liquid as soon as the precipitate settles.

Ferric acetate or chlorid solution.

Alkanna tincture, diluted with an equal bulk of water.

Aqueous solution of safranin.

Hydrochloric acid (10 per cent).

Acetic acid.

## (c) PREPARATION OF THE MATERIAL.

Reduce a portion of the sample to a fine powder in a mortar. Separate another portion into several grades of fineness in sieves of different mesh or by jarring on a sheet of paper. In the coarser grades fragments of a suspicious nature may often be seen with the naked eye or under a simple microscope, which should be picked out with forceps for subsequent examination under the compound microscope.

## (d) EXAMINATION.

Mount a small amount of the ground sample in water and examine under the compound microscope with both ordinary and polarized light. This gives a general insight into the nature of the material and serves for the detection and identification of starch granules and various tissues.

Draw a small drop of iodine solution into the same preparation by means of a piece of filter paper placed on the opposite edge of the cover glass and examine. Starch granules will be colored blue or blue-black, cellulose yellow, and proteids either brown or yellow.

In the manner described draw a little potassium hydroxid solution under the cover glass and examine once again. This treatment gelatinizes the starch granules, dissolves the proteids, saponifies the fats, and in other ways clears the preparation. It also imparts to tannins a reddish color.

If treatment with potash does not clear the tissues satisfactorily, treat a fresh portion for some hours with chloral hydrate solution.

Examine also the crude fiber obtained in the chemical analysis, as in this material the stone cells and other tissues are beautifully distinct. <sup>a</sup>

To isolate stone cells, bast fibers, and other thick-walled cells macerate a portion of the sample in Schultze's liquid, using such proportion of potassium chlorate and

nitric acid and heating for such a time as secures the desired results. Powdered charcoal and charred shells resist the bleaching action of potash, chloral hydrate, and Schultze's liquid, the fragments after, as before the treatment, being black and opaque.

If it is desired to distinguish cellulose from infiltrated substances (lignin, suberin, etc.), add to a water mount freshly prepared chlorzinc iodine, which colors the former blue and the latter yellow.

As a test for proteids, cautiously warm, on a slide, with a drop of freshly prepared Millon's reagent. The proteids are partially disorganized, taking on gradually a brick-red color. If it is desirable to study the form of the aleurone (proteid) granules, which in some plants are quite as characteristic as starch granules, prepare a mount in pure glycerin or oil.

To distinguish fats, oils, essential oils, and resins from other cell contents, treat for an hour with alkanna tincture diluted with an equal bulk of water, which imparts to these substances a deep red color; or treat with ether, which dissolves them. Treat also with alcohol, which dissolves the essential oils and resins but does not perceptibly affect the fats and oils.

In testing for tannins and tissues impregnated with those substances, add ferric acetate or chlorid solution. Both of these reagents give with tannins a green or blue color, but the former acts more slowly and is to be preferred.

Crystals of calcium oxalate are recognized by their characteristic forms and their deportment with polarized light. To distinguish calcium oxalate from calcium carbonate, treat with acetic acid, which does not affect the former but dissolves the latter with effervescence. Both are soluble in hydrochloric acid.

Other special reagents may be occasionally useful, but as a rule, reagents play a subordinate part in the microscopic examination of spices, the chief factor being a thorough understanding of the size, shape, color, and other characteristics of the histological elements, which can be learned only by experience.

## XI.—VINEGAR.

By WM. FREAR,

*Chemist of State Experiment Station, State College, Pa.*

Vinegars may be *defective* because of the poor quality of the liquids from which they are prepared or because of incomplete fermentation; *impure* from the development of abnormal fermentations, invasion of vinegar eels (*Anguillula oxophila*), moulds, or presence of foreign substances accidentally introduced, such as metallic salts formed by the action of the acid upon metallic vessels, faucets, etc.; *adulterated*, as by dilution, addition of mineral acids, pungent materials, coloring matters, etc.; *misbranded*, as when a pure vinegar of one sort is sold under the name of another. It is needful that methods for the detection of all the foregoing classes of variations shall be employed as occasion demands; that is, variation in normal constituents as well as presence of foreign matters must be detected and often measured.

### 1.—PREPARATION OF SAMPLES.

For microscopic examination, the original sample should be employed, but for chemical analysis the vinegar should be filtered, if turbid from the presence of suspended solids. Samples should be kept in glass bottles with ground-glass stoppers and should be promptly analyzed to avoid fermentative changes, which progress rapidly in the warm air of the laboratory.

### 2.—CALCULATION OF RESULTS.

Express all results as per cent by weight. Owing to the slight departure of the specific gravity of vinegars from that of water, no error of importance in most con-

trol work arises from considering the weight of one cubic centimeter of vinegar to be one gram. Where greater accuracy is desired, the quantities of vinegar employed may be accurately weighed, or the weight estimated from a direct determination of the specific gravity, or the average specific gravity of the kind of vinegar used may be taken as a means of a closer approximation than that attained by regarding the specific gravity of the vinegar to be 1.000. Blyth<sup>a</sup> gives the following figures:

	Range.	Average.
Wine vinegar .....	1. 0129-1. 0213	1. 0175
Spirit vinegar .....	1. 008-1. 013	1. 0082
Malt vinegar .....	.....	1. 0150
Sugar vinegar .....	.....	1. 0100
Wood vinegar .....	.....	1. 0070

The writer has found in the case of cider vinegars a range of 0.997 to 1.029, with an average of 1.015.

### 3.—DETERMINATION OF SPECIFIC GRAVITY.

Proceed as directed under wines (p. 82).

### 4.—DETERMINATION OF TOTAL SOLIDS OR EXTRACT.

Evaporate 10 cc in a tared platinum dish of 50-mm diameter on the steam bath to a sirupy consistency, dry for 2½ hours in the drying oven at the temperature of boiling water, cool and weigh.

### 5.—DETERMINATION OF ASH.

Use the method employed in case of wines (p. 83).

### 6.—DETERMINATION OF SOLUBILITY, ALKALINITY, AND PHOSPHORIC ACID OF ASH.

Smith's method, modified by Frear:<sup>b</sup> Evaporate 25 cc to dryness, burn, cool, weigh; extract the ash repeatedly with hot water on an ash-free filter; dry and ignite the filter with undissolved residue, cool and weigh, and calculate as insoluble ash. Titrate the aqueous extract with decinormal acid, using methyl orange as indicator. Acidulate the neutralized solution with nitric acid; also treat the insoluble ash with nitric acid, and precipitate the phosphoric acid from the two solutions separately by molybdate solution. The yellow precipitates, after solution in ammonia, may either be treated with magnesia mixture, as in ordinary ash analysis, or reduced with zinc and sulphuric acid, and titrated with standard permanganate solution. Express the results in terms of milligrams of  $P_2O_5$  in the water-soluble and water-insoluble ash, respectively, from 100 grams of vinegar.

### 7.—DETERMINATION OF TOTAL ACIDITY.

Dilute 12 cc in a beaker until the solution appears very slightly colored when viewed against a white background, add a few drops of phenolphthalein indicator, and titrate with half-normal sodium hydroxid. Multiply the number of cubic centimeters of half-normal soda solution required by 0.03 for the total acidity expressed as grams of acetic acid. When 12 grams of vinegar are employed in the determination, the number of cubic centimeters of half-normal alkali employed may be divided by 4 for the percentage of total acids expressed in terms of acetic acid.

<sup>a</sup>Foods: Their comp. and anal., 4th ed., p. 582.

<sup>b</sup>Jour. Am. Chem. Soc., 1898, 20, 5.

## 8.—DETERMINATION OF VOLATILE AND FIXED ACIDS.

## (a) VOLATILE ACIDS.

Heat 15 cc of the vinegar to boiling in a flask, adding a little tannin if foaming occurs; then lower the flame and pass a current of steam through the vinegar to a condenser. Continue the operation until 15 cc of distillate shows no acidity upon a test with sensitive litmus paper. Titrate the combined distillate with half-normal sodium hydroxid, using phenolphthalein as indicator. The number of cubic centimeters of alkali required, multiplied by 0.03, gives the weight of volatile acids, expressed as grams of acetic acid.

## (b) FIXED ACIDS.

Deduct volatile acids from total acids and multiply the remainder by 0.817 for sulphuric acid, or 1.117 for malic acid. Or dilute the nonvolatile residue from the distillation with water until the solution appears nearly clear against a white background. Titrate with half-normal sodium hydroxid, using phenolphthalein as indicator, as in case of the volatile acids. The weight of fixed acids, calculated to sulphuric or malic, is calculated by the factors given above. When 15 cc are taken, multiply the number of cubic centimeters of half-normal alkali solution employed by 0.163 for the percentage of fixed acids expressed in terms of sulphuric acid ( $\text{H}_2\text{SO}_4$ ), or by 0.223 to express in terms of malic acid.

## 9.—DETECTION OF FREE MINERAL ACIDS.

## (a) FIRST METHOD.\*

Prepare an extract of logwood by pouring 100 cc of boiling water upon 2 grams of fresh logwood chips, allowing the decoction to stand for a few hours and filtering. Separate drops are spotted on a porcelain surface and dried over a water or steam bath. Add to one of the spots a drop of the vinegar to be tested (after concentration, if thought desirable); again evaporate to dryness. A yellow tint remains if free mineral acids are absent, a red tint if present.

## (b) SECOND METHOD.

To 5 cc of vinegar add 5 or 10 cc of water; after mixing well, add 4 or 5 drops of an aqueous solution of methyl-violet (one part of methyl-violet 2B in 10,000 parts of water). The occurrence of a blue or green color indicates the presence of a free mineral acid.

## 10.—DETERMINATION OF FREE MINERAL ACIDS.

## (a) HILGER'S METHOD.

Neutralize 20 cc of the vinegar exactly with half normal alkali, the end reaction being determined by the action of drops of the liquid upon sensitive violet litmus paper. Evaporate the neutral liquid to one-tenth volume in a porcelain dish, add a few drops of methyl-violet solution (that mentioned in paragraph 9), dilute with 3 or 4 cc of water, if needful, to secure a clear solution, bring to boiling, and titrate with half normal sulphuric acid till a green or blue color begins to appear. The difference, in cubic centimeters, between the seminormal alkaline and acid reagents added, multiplied by the factor 0.1225, expresses the percentage of mineral acid present, in terms of sulphuric acid ( $\text{H}_2\text{SO}_4$ ).

## (b) HEHNER'S METHOD.

To a weighed quantity of the sample add excess of decinormal alkali, evaporate to dryness, incinerate and titrate the ash with decinormal acid. The difference between

\* Ashby, Allen's Com. Org. Anal. 2d ed., vol. I, p. 393.



the number of cubic centimeters of alkali added in the first place and the number of cubic centimeters needed to titrate the ash represents the equivalent of the free acid present.

#### 11.—DETERMINATION OF OXALIC ACID.

The presence of oxalic acid may be detected and its quantity determined by addition of a solution of calcium sulphate to a measured quantity of the vinegar.

#### 12.—DETERMINATION OF ALCOHOL.

Domestic fruit vinegars are often incompletely acetified; if the specific gravity be abnormally low, a determination of the alcohol present is desirable. Owing to the small quantities usually present, it is best to concentrate the distillates. Carefully neutralize 100 cc of the vinegar and distill over 40 cc; redistill the distillate until 20 cc has passed over; cool to 15.5° C. and make up to 20 cc with distilled water. Determine the specific gravity by means of a pycnometer and calculate the percentage by weight, by Table II. The percentage in the last distillate, divided by 5, represents the amount in the original vinegar.

#### 13.—DETECTION OF COLORING MATTERS.

The principal coloring matter used for tinting imitation vinegars in America is caramel. To detect this use Amthor's method (p. 120). A further test of the caramel may be made by boiling the aqueous solution of a portion of the precipitate obtained by Amthor's method, with Fehling's solution; caramel has a reducing action.

In the case of wine vinegars, test for foreign red colors may be made according to the methods given on p. 111 and following.

#### 14.—DETECTION OF FOREIGN PUNGENT MATERIALS.

Exactly neutralize a portion of the vinegar (the residue from determination of total acidity may be used), evaporate off a portion of the water, and test the concentrated solution by taste for pungent substances; then agitate the liquid with ether, in a separatory funnel, remove and evaporate the ethereal layer, and apply the same test to the residual ethereal extract. The perfect identification of the specific substance employed is rarely attained.

#### 15.—DETECTION AND DETERMINATION OF METALLIC POISONS.

Evaporate from 200 to 500 cc to dryness. In case of cider, malt, and other vinegars rich in solids, add a little sodium hydroxid and potassium nitrate, and incinerate. The ash thus obtained, or the solids themselves, in case of vinegars low in extract, is carefully dissolved in hydrochloric acid and the solution subjected to examination by methods indicated for the analysis of canned vegetables (p. 52).

#### 16.—DETECTION OF PRESERVATIVES.

Preservatives are sometimes, though rarely, added to vinegar. Salicylic and benzoic acids may be separated by agitation with ether and detected in the residue left upon evaporation of the ether. Boric acid may be detected in the ash left upon the evaporation and incineration of the vinegar after neutralization by alkali.

Most of the tests for the detection of formaldehyde are excluded from use in testing vinegars, since the indicative reactions are usually produced by acetaldehyde also, which is frequently present in normal vinegar. From 100 cc of the vinegar distill 20 cc. To a portion of the distillate add a few drops of milk; float the liquid upon 90 to 94 per cent sulphuric acid to which a little ferric chlorid has previously been added. A violet ring appears at the point of separation of the liquids, if formaldehyde is present. Acetaldehyde does not produce this reaction, the color being yellowish green changing to brown.

A portion of the distillate prepared as above may, after the addition of a few drops of milk, be treated with a drop of concentrated aqueous ferric chlorid solution, agitated and well mixed with a nearly equal volume of concentrated hydrochloric acid. Warm below boiling with constant agitation. Shortly before ebullition a purple coloration of the casein appears, if formaldehyde be present; it is not produced by acetaldehyde.

#### 17.—DETERMINATION OF THE SOURCE OF A VINEGAR.

It is not always possible to make this distinction with entire certainty. The principal vinegar of the United States is cider vinegar, though malt vinegar finds preference with many. The substitutes are chiefly (a) low wine vinegar, either sold under the name "white wine vinegar" or colored by addition of caramel, or even given color and body by addition of cheap apple jelly; (b) vinegar from sugarhouse wastes; (c) wood vinegar, or preparations from vinegar essence, with or without coloring matters. Grape, or true "wine vinegar," is important in few localities outside of California. Glucose vinegar is sometimes found.

The nature of the vinegar is commonly indicated, if it be pure of its kind, by its flavor and odor. The fruity quality of cider vinegar is usually very conspicuous; the odor of malt vinegar is characteristic; and impure wood vinegar often shows a very perceptible empyreumatic quality. Even when these qualities are distinctly indicative of the source of the vinegar, additional evidence is desirable for legal proof; often slight impurities mask them.

The *quantity of the solids* is often distinctive. The range for the principal vinegars is: Cider vinegar, 1.18 to 8.04; average, about 2.5. Malt vinegar, 1.75 to 6.0; average, about 3.0. Spirit vinegar, 0.13 to 0.78; average, about 0.3. Wine vinegar, 1.38 to 3.19; average, 1.9.<sup>a</sup> The quantity of solids in sugar and glucose vinegars varies with the conditions of manufacture, sometimes corresponding closely with that of fruit vinegars. That of wood vinegar resembles the quantity in spirit vinegars. By the addition of foreign solids to spirit or wood vinegar the value of this criterion is often destroyed.

The *quality of the vinegar solids*, as a whole, is usually characteristic. The *consistency* of that from cider is thick and viscid or mucilaginous; that from sugar, glucose, or malt is somewhat more glutinous. The *odor* of baked apples is notable in cider-vinegar solids, that of molasses is often apparent in sugar-house vinegar, and that of malt vinegar is usually distinctive. The *flavor* of cider-vinegar solids is acid and somewhat astringent; in these respects wine vinegar resembles it. The bitter taste of caramel is usually observed in sugar-house vinegar solids and in those from colored spirit and wood vinegars. On burning the solids, the apple odor is developed by cider vinegar, that of burnt sugar by sugar-house vinegar, and that of scorched corn<sup>b</sup> by glucose vinegar. The *solubility of the solids in alcohol* marks fruit vinegars—except a granular residue of tartar in grape vinegar—while the solids of malt and glucose vinegars are only very slightly dissolved.<sup>c</sup> By addition of cheap cider jelly to spirit or wood vinegar, the characteristic apple quality is, however, given to the vinegar solids.<sup>d</sup>

The *quantity of the ash* is useful in distinguishing spirit and wood vinegars from fruit and malt vinegars, the quantity in the former case rarely exceeding 0.1 per cent; in the latter rarely falling below 0.2; the range for pure cider vinegar is 0.19 to 0.57; average about 0.35 per cent.

The *quality of the ash* is far more indicative. That of fruit vinegars and malt vinegars is distinctly alkaline; that of spirit and wood vinegars very slightly so. The

<sup>a</sup> According to Blyth. Eckenroth gives 0.35 to 1.51 per cent.

<sup>b</sup> Davenport, 26th Ann. Rept. Milk Inspector, City of Boston, 1885.

<sup>c</sup> Allen, Com. Organic Anal., Vol. I, p. 389.

<sup>d</sup> Frear, Rept. Pa. Dept. of Agr., 1898, p. 138; Leach, Rept. Mass. State Bd. of Health, 1898, p. 633.

*alkalinity*<sup>a</sup> expressed in terms indicated in the previously described methods is, for cider vinegar 26 to 65, average 39; for malt vinegar 5.5, and for spirit vinegar 1.1. The quantity of phosphoric acid in the ash, expressed in milligrams per 100 grams of the vinegar, ranges in case of pure cider vinegar from 9 to 39; in malt vinegar it is variously stated to range from 9 to 12.5 and from 50 to 100; while the ash of spirit vinegar shows traces only. The solubility of the phosphoric acid in cider vinegar is high, 50 to 75 per cent of the amount present being soluble in water, or from 4.7 to 22.7 mg per 100 grams of vinegar; no soluble phosphoric acid is found in the ash of spirit vinegar. If cider vinegars be diluted with hard water, the relatively insoluble earthy phosphates are formed and the proportion of soluble phosphoric acid greatly reduced.

The flame reaction obtained by igniting a drop of the solids upon a loop of platinum wire in a colorless flame is, in case of cider vinegar, exclusively that of potash; solids obtained from artificially colored vinegars, sugar, spirit, and glucose vinegars always give the sodium flame.<sup>b</sup>

The optical activity of vinegars is often characteristic. For this purpose they should be examined in a 400-mm tube, after treatment with lead subacetate or boneblack, if necessary, for their clarification. Pure cider vinegar is slightly levorotatory, ranging from 0.96° to 2.79° Ventzke, in thoroughly acetified samples. Vinegar containing unfermented apple solids will be much more highly levorotatory; wine vinegar is also slightly levorotatory. On the other hand, vinegar from sugarhouse wastes is dextrorotatory before and levorotatory after inversion; that from glucose dextrorotatory both before and after inversion.<sup>c</sup> Incompletely acetified vinegar made from incompletely fermented cider might exhibit the optical properties of a mixture of spirit vinegar and cider jelly, but the latter would be distinguished by differences at other points.

The ratio of reducing sugars, after inversion, to total solids is a means of distinguishing genuine cider vinegar from spirit vinegar supplied with solids from cider jelly. Where they constitute 50 per cent or more of the solids, the vinegar may be regarded as not pure cider vinegar. An incompletely fermented cider which had begun acetification might be mistaken for the imitation product if this ratio alone were considered; but low acetic acid, high alcohol, and other characters, make it easy to distinguish the former. The determination should be made as follows: Pipette 25 cc of the vinegar into a 100-cc flask; add 5 cc of concentrated hydrochloric acid; heat the flask in a water bath at 70° C., and after the vinegar has attained a temperature of 67°, maintain it at 67° to 70° for five minutes; then cool. Dilute one half; neutralize exactly with sodium hydroxid solution, using phenolphthalein as an indicator; make up to 100 cc and withdraw 25 cc for determination of reducing sugar by the copper-reduction method of Allihn, computing the results by the table of Meissl and Wein.<sup>d</sup> In case a large ratio of reducing sugar to total solids be found, its origin—whether from cider jelly, sugarhouse wastes, or glucose—remains to be determined, either by collateral evidence or by a more detailed separation of the several sugars in the solids. For the latter purpose, employ the methods described by Browne.<sup>e</sup>

The ratio of ash to solids is also indicative. In cider vinegar the ratio ranges from

<sup>a</sup> Smith, A. W., Jour. Am. Chem. Soc., 1898, 20, 7; Blyth, Foods: Their Comp. and Anal., 4th ed., p. 587; Hehner, Vjschr. Chem., Nahr, 1893, 7, 194.

<sup>b</sup> Davenport, 26th Ann. Rept. Milk Inspector, City of Boston, 1885.

<sup>c</sup> Frear, Rept. Pa. of Dept. of Agr., 1898, p. 145; C. A. Browne, jr., Rept. Pa. State College Agr. Exp. Station, 1900, p. 265; also Bulletin 58, Pa. Dept. of Agr., p. 43; Doolittle & Hess, Jour. Am. Chem. Soc., 1900, 22, 19; Allen, Com'l Organic Analysis, second edition, Vol. I, p. 81; Von Bitteryst, Rev. fals. internat., 1894, 7, 151.

<sup>d</sup> Wiley, Agricultural Analysis, Vol. III, p. 159.

<sup>e</sup> Rept. Pa. State College Agr. Expt. Sta., 1900, p. 269-274; Cf. also, Frear, Rept. Pa. Dept. of Agr., 1898, p. 138 and following, and Browne, Bul. 58, Pa. Dept. of Agr., for comparative data.

4.6 to 17.1, average 9.0; that of pure spirit—malt and wine vinegar—averages 5 to 8; but upon addition of cider jelly to a vinegar, low in solids, the ratio becomes 17.1 to 80.

The quantity of nitrogen serves to distinguish malt vinegars from such as are derived from saccharine liquids, low wines, or wood acids. Calculated as albuminoids, the amount in malt vinegars is 0.65 to 0.7 per cent;<sup>a</sup> in cider vinegars, 0.006 to 0.024 per cent;<sup>b</sup> in sugar, glucose, spirit, and wood vinegars, much less.

The presence of alcohol in vinegars derived from alcoholic liquids often serves to distinguish them from wood vinegar; its absence is not conclusive against their derivation from the former class of materials.

The presence of tartar distinguishes wine vinegar, though its absence is not conclusive of other origin.<sup>c</sup> Allen's method for this test is as follows:<sup>d</sup> Treat the residue left from evaporation of the vinegar, with alcohol; a granular residue of tartar remains undissolved; to prove its character, pour off the alcohol and dissolve the residue in a small quantity of hot water. On cooling the aqueous solution, and stirring the sides of the vessel with a glass rod, the acid tartrate of potassium will be deposited in streaks on the track of the rod. An addition of an equal bulk of alcohol makes the reaction more delicate.

The adulteration of wine vinegar by addition of free tartaric acid is proved in a similar manner, the alcoholic solution of the extract is treated with an alcoholic solution of potassium acetate; upon stirring the mixture with a glass rod in a beaker, streaks and probably a distinct precipitate of tartar will be deposited. Quantitative results can be obtained upon titration of the precipitate by standard alkali.

The presence of malic acid distinguishes cider vinegars, though the quantity is often small. Failure to obtain a precipitate upon the addition of a few drops of neutral lead acetate to 10 cc of a vinegar, proves it not to be cider vinegar; if a precipitate be obtained, parallel tests with silver nitrate and barium chlorid to determine the absence of chlorids and sulphates should be made, before the presence of malic acid be considered proved.

Dextrin is often found in glucose vinegar and is precipitated from the concentrated vinegar upon addition of three or four volumes of strong alcohol; the precipitate may be identified by the physical properties and by its color reaction with iodine solution. Dextrin is also of general occurrence in malt vinegar.

Wood vinegar is quite commonly marked by the presence of *emphyreumatic matters*; these are sometimes sufficient to impart their characteristic flavor to the vinegar. It has been recommended that the method of Cazeneuve and Cotton<sup>e</sup> be used for their detection; this depends upon the immediate reduction of 1 cc of a 0.1 per cent solution of potassium permanganate when added to 10 cc of the liquid to be tested. Obviously this test is not applicable in the presence of caramel or the reducing sugars of fruit and malt vinegars; both the distillate and the ether extract of cider vinegars cause rapid reduction. The test is not applicable therefore to mixtures of wood vinegar with that from other sources, but may be useful in completing the examination of a vinegar shown by other evidence to be wood vinegar.

Microscopic examination may establish the absence of alcoholic and acetic ferments; in such event, the article is shown to be distilled vinegar.

<sup>a</sup> Blyth, Foods, Their Comp. and Anal., 4th edition, p. 587.

<sup>b</sup> Frear, Rept. Pa. Dept. of Agr., 1898, p. 145.

<sup>c</sup> Tretzol (*Forschungsber.*, 1896, 3, 186; *Vjschr. Chem. Nahr.*, 1898, 11, 257) states that true wine vinegars are occasionally found without tartar or more than traces of it. H. Eckenroth (*Pharm. Ztg.*, 1889, 34, 14; *Vjschr. Chem. Nahr.*, 1891, 4, 88) claims that it is always found, if from 500 cc to 1 liter of the vinegar be used for the test. Von Bitteryst (*Op. cit.*, 1896, 9, 425) gives 0.04 gram per 100 cc as a minimum.

<sup>d</sup> Commercial Organic Analysis, 2d ed., vol. 1, p. 389.

<sup>e</sup> Vereinbarungen zur einheitlichen Untersuchung und Beurtheilung von Nahrungs- und Genussmitteln, II, 83. *Bul. soc. chim.*, 1881, [2], 35, 102.

**XII.—FLAVORING EXTRACTS.**

By A. S. MITCHELL,

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Flavoring extracts consist of three classes of preparations—tinctures, spirits, and artificial essences. Each class needs specific treatment, varying in detail with the object sought. In quantitative examination of either tincture of vanilla or spirits of lemon, it is essential to carry out comparative tests upon similar products of known purity and strength. Many of the methods recommended for lemon may be readily adapted to the examination of other spirits containing volatile oils.

**(A) VANILLA AND ITS SUBSTITUTES.****1.—DETERMINATION OF TOTAL SOLIDS.**

Weigh 25 grams of the extract into a large, flat-bottomed dish which contains enough freshly ignited asbestos to absorb it; dry from twenty to twenty-four hours in a water-jacketed oven.

**2.—DETERMINATION OF ASH.**

Weigh about 10 grams of the extract in a flat-bottomed platinum dish, evaporate to dryness on a water bath, heat slowly until intumescence ceases, and ignite in a muffle at a low red heat until a white ash is obtained.

**3.—EXAMINATION OF ASH.**

Proceed as directed on page 106.

**4.—DETECTION AND DETERMINATION OF COUMARIN AND VANILLIN.<sup>a</sup>**

Dealcoholize 50 grams of the extract in a glass evaporating dish upon a water bath at a temperature of about 80° C.; add water from time to time to retain the original volume. After removal of the alcohol, add normal lead acetate solution, drop by drop, until no more precipitate forms. Stir with a glass rod to facilitate flocculation of the precipitate. Filter through a moistened filter; wash three times with a few cubic centimeters of hot water. Cool the filtrate and extract with ether by shaking out in a separatory funnel. Use 15 to 20 cc of ether at each separation and repeat the shaking out three or four times, or until a few drops of the ether evaporated upon a watch glass leaves no residue. Place the combined ether extracts containing all of the vanillin and coumarin in a clean separatory funnel and shake out repeatedly with from 5 to 10 cc of 7 per cent ammonia.<sup>b</sup> Repeat the treatment with ammonia once or twice after the fractions cease to be colored yellow.

Set aside the combined ammoniacal solutions for the determination of vanillin.

Wash the ether solution into an evaporating dish and allow the ether to evaporate at ordinary temperature. Extract the residue by treating it at room temperature with 5 to 10 cc of a petroleum fraction boiling between 30° and 40° C.; allow it to stand several minutes and then decant into a dried, weighed evaporating dish. Repeat the extraction with petroleum ether until a drop evaporated on a watch glass leaves no residue. Allow the petroleum ether to evaporate at room temperature; dry in a desiccator over sulphuric acid and weigh as coumarin.

The residue should be crystalline and have a melting point of 67° C. This, with the characteristic odor of coumarin, obtained by warming slightly, is sufficient for its identification.

<sup>a</sup> Hess and Prescott, Jour. Am. Chem. Soc., 1899, 21, 256.

<sup>b</sup> Winton prefers 2 per cent ammonia.

Slightly acidulate the ammoniacal solution reserved for vanillin with 10 per cent hydrochloric acid. Cool and shake out in a separatory funnel with 10 cc of chloroform, repeating the process as described for ether extraction. Evaporate the chloroform at room temperature and dry over sulphuric acid, or in an air bath at a temperature not exceeding 55°.

Wash the residue with boiling petroleum ether, using 5 to 10 cc at each treatment; decant the fractions into a dry, weighed evaporating dish. Evaporate the petroleum ether at room temperature and dry as before. Weigh the residue as vanillin.<sup>a</sup> This should be pure and crystalline and have a melting point of 80° to 81°. This, with the characteristic odor and crystalline form, is sufficient for its identification as vanillin.

#### 5.—DISTINCTION OF TRUE EXTRACT OF VANILLA FROM LIQUID PREPARATIONS OF VANILLIN.<sup>b</sup>

##### (a) THEORY.

The leading fragrant principle of the vanilla bean and of true vanilla extract is vanillin, a definite chemical compound—hydroxymethoxybenzoic aldehyde. It is not the only fragrant or valuable constituent of vanilla bean and true vanilla extracts. The artificial vanillin, made for the market, contains vanillin identical with the vanillin contained in the vanilla bean; but the vanilla bean, as the vanilla extract, contains among its many "extractive matters" which enter into the food and fragrant value of vanilla extract, certain resins which can be identified with certainty in analysis by a number of determining reactions. Extract made without true vanilla can be detected by negative results in all these reactions.

Vanilla beans contain 4 to 11 per cent of this resin. It is of a dark red to brown color and furnishes about one-half the color of the extract of vanilla. This resin is soluble in 50 per cent alcohol, so that in extracts of high grade, where sufficient alcohol is used, all resin is kept in solution. In cheap extracts, where as little as 20 per cent of alcohol by volume is sometimes used, an alkali—usually potassium bicarbonate—is added to aid in getting resin, gums, etc., in solution, and to prevent subsequent turbidity. This treatment deepens the color very materially.

##### (b) METHOD OF ANALYSIS.

Place 50 cc of the extract to be examined in a glass evaporating dish and evaporate the alcohol on the water bath. When alcohol is removed, make up about the original volume with hot water. If alkali has not been used in the manufacture of the extract, the resin will appear as a flocculent red to brown residue. Acidify with acetic acid to free resin from bases, separating the whole of the resin and leaving a partly decolorized, clear supernatant liquid after standing a short time. Collect the resin on a filter, wash with water, and reserve the filtrate for further tests.

Place a portion of the filter with the attached resin in a few cubic centimeters of dilute caustic potash. The resin is dissolved to a deep red solution. Acidify. The resin is thereby precipitated.

Dissolve a portion of the resin in alcohol; to one fraction add a few drops of ferric chlorid; no striking coloration is produced. To another portion add hydrochloric acid; again there is little change in color. In alcoholic solution most resins give color reactions with ferric chlorid or hydrochloric acid.

To a portion of the filtrate obtained above add a few drops of basic lead acetate. The precipitate is so bulky as to almost solidify, due to the excessive amount of organic acids, gums, and other extractive matter. The filtrate from this precipitate is nearly, but not quite, colorless.

<sup>a</sup>See Appendix, p. 155.

<sup>b</sup>Jour. Am. Chem. Soc., 1899, 21, 719.

Test another portion of the filtrate from the resin for tannin with a solution of gelatin. Tannin is present in varying but small quantities. It should not be present in great excess.

#### 6.—DETERMINATION OF CANE SUGAR.

See methods given on page 85.

#### 7.—DETERMINATION OF ALCOHOL.

Proceed as directed on page 82.

#### 8.—TESTS FOR COLORING MATTER—CARAMEL.

Of the artificial colors used in vanilla extracts, the most common is caramel.

*Preliminary test.*—If on shaking the bottle of vanilla the bubbles formed are of a bright caramel color, and they keep this color until the very last are gone, it indicates presence of caramel. This difference is readily shown by comparison with known pure samples.

*Lead acetate test.*—The coloring matter present in vanilla, or tonka, extracts is almost completely removed when the dealcoholized extract is treated with a few cubic centimeters of basic lead acetate solution. When caramel is present, the filtrate and precipitate, if any, have the characteristic red-brown color of caramel.

*Phenylhydrazin hydrochlorid test.*—To 20 cc of the extract add 1 cc of zinc chlorid (5 per cent solution), and then add 1 cc of a 2 per cent solution of potassium hydroxid and stir the precipitate with the solution. Filter and wash the precipitate with hot water. Dissolve the precipitate in 10 cc or more of a 10 per cent solution of hot acetic acid, receiving this solution in an evaporating dish; evaporate to 5 cc over water bath. Neutralize the excess of acetic acid with potassium hydroxid solution and divide the solution equally in two 6-inch test tubes. Add to each 5 to 10 cc of a solution of phenylhydrazin hydrochlorid and sodium acetate, prepared by dissolving 2 grams of phenylhydrazin and 3 grams of sodium acetate in 15 cc of water. Let one tube stand overnight. Heat the other on a water bath for half an hour. A brown precipitate will be obtained in both cases if caramel be present.

Fullers' earth may be substituted for zinc hydroxid. In such case, stir or shake a small amount of the earth with the extract, filter and wash with cold water. Extract the caramel with boiling water. Test the concentrated water solution with phenylhydrazin as above.

#### (B) LEMON EXTRACT.

These extracts are made by the solution of oil of lemon or its more soluble constituents in alcohol of varying strength. Oil of lemon contains nearly 90 per cent of terpenes, largely d-limonene, giving the oil a rotary power of 59 to 64 circular degrees at 20° F. The aromatic constituents are oxygenated bodies, the principal of which is citral. The latter bodies being more soluble in weak alcohol frequently constitute the chief flavoring substance present in pure extracts of the cheaper quality.

Extracts of the highest strength and purity are made by solution of the whole oil of lemon in deodorized alcohol, as described in the United States Pharmacopoeia. The lowest quality of adulterated extracts may contain minute amounts of citronella aldehyde or citral obtained from "lemon grass" (*Andropogon citratus*), together with aromatic or pungent tinctures, such as mace or capsicum.\*

#### 1.—DETERMINATION OF TOTAL RESIDUE.

Evaporate 10 grams of the extract on a water bath at a temperature below the boiling point of the alcohol. In the absence of glycerol dry to a constant weight at

\*Dr. William Frear has suggested that if a few drops of bromin water are added to 3 cc of true lemon extract, the bromin will be almost instantaneously absorbed by the terpene present.

100° C. Examine the residue for sugar and glycerol. The presence of capsicum may be readily detected by taste.

## 2.—DETERMINATION OF GLYCEROL.

Proceed as directed on page 82.

## 3.—DETERMINATION OF ASH.

Ignite the residue from 10 grams of the extract at a dull red heat. Examine the ash for magnesia. Where the extract has been made with insufficient alcohol to effect complete solution of the oil, the liquid is frequently clarified by filtration with magnesia; in which case the latter substance may be detected in the ash.

## 4.—DETERMINATION OF SPECIFIC GRAVITY.

Determine the specific gravity as directed on page 82.

## 5.—DETERMINATION OF ALCOHOL.

(a) Dilute 50 cc to 200 cc; pour mixture into dry Erlenmeyer flask containing 5 grams of light carbonate of magnesia. Stopper, shake well, and filter quickly through a large, dry, plaited filter. Determine the alcohol in 150 cc of the filtrate, as directed on page 82, multiplying the results so obtained by  $\frac{4}{3}$  to correct for the aliquot part taken.

(b) In the absence of appreciable quantities of solids or glycerol, calculate the alcohol approximately from the specific gravity of the extract, using Table II.

## 6.—DETECTION OF METHYL ALCOHOL.

These methods consist in the conversion of the alcohols to aldehydes by the method of Mulliken and Scudder,<sup>a</sup> the removal of acetaldehyde, and the detection of formaldehyde when produced.

Dilute a portion of the distillate obtained in the determination of alcohol (or, for preliminary examination, dilute and filter the original extract) until the liquid contains approximately 12 per cent of alcohol by weight.

Oxidize 10 cc of the liquid in a test tube as follows: Wind copper wire 1 mm thick upon a rod or pencil 7 to 8 mm thick in such a manner as to inclose the fixed end of the wire and to form a close coil 3 to 3.5 cm long. Twist the two ends of the wire into a stem 20 cm long and bend the stem at right angles about 6 cm from the free end, or so that the coil may be plunged to the bottom of a test tube, preferably about 16 mm wide and 16 cm long. Heat the coil in the upper or oxidizing flame of a Bunsen burner to a red heat throughout. Plunge the heated coil to the bottom of the test tube containing the diluted alcohol. Withdraw the coil after a second's time and dip it in water. Repeat the operation from three to five times, or until the film of copper oxide ceases to be reduced. Cool the liquid in the test tube meanwhile by immersion in water. Remove 10 drops of the liquid so oxidized to a small porcelain capsule and reserve this for a separate test.

### (a) REMOVAL OF THE ACETALDEHYDE BY PRESCOTT'S METHOD.<sup>b</sup>

Add to the liquid remaining in the test tube 6 cc of a 3 per cent solution of hydrogen peroxid or an equivalent amount. Mix and filter into a porcelain dish. After three minutes add 2 cc of a 10 per cent solution of sodium thiosulphate. After two or three minutes place the dish in a good white light and test for formaldehyde.

*Test for formaldehyde.*—To the contents of the dish add 3 cc of a phloroglucin solu-

<sup>a</sup> S. P. Mulliken and H. Scudder, Amer. Chem. Jour., 1899, 21, 266.

<sup>b</sup> Pharmaceutical Archives, 1901, vol. 4, No. 5.



tion made by dissolving 1 gram of phloroglucin and 20 grams of sodium hydroxid in sufficient water to make 100 cc. A bright red coloration (not purple) indicates the presence of methyl alcohol in the original sample. When too little hydrogen peroxid is added an orange-yellow color will slowly appear. The hydrogen peroxid, if not fully destroyed, will give rise to a purple color of gradual formation. The cherry or raspberry red produced as a result of methyl alcohol appears quickly after the addition of the reagent, and fades quickly unless quite intense. The intensity of the red color is in proportion to the quantity of methyl alcohol present. If the wood alcohol be as much as 1 part to 20 of ethyl alcohol, its presence will be revealed by this test.

For comparison, test the ten drops reserved in the casserole for formaldehyde after the addition of 10 cc of milk by Leach's modification of Hehner's test as directed on page 108, using care not to boil the mixture.<sup>a</sup>

(b) REMOVAL OF FORMALDEHYDE BY METHOD OF S. P. MULLIKEN.<sup>b</sup>

Oxidize 5 cc of the diluted alcohol as directed above. Add 1 cc of strong ammonia to the oxidized liquid in a casserole and expel the acetaldehyde by boiling gently over a direct flame until the vapor ceases to smell of ammonia. Add 2 to 3 drops of strong hydrochloric acid to set free the formaldehyde which has been retained as hexamethyltetramin, and bring the liquid momentarily to a boil; cool promptly by immersion of the casserole in water and test for formaldehyde by the modified resorcin test,<sup>c</sup> as follows:

Add to the liquid remaining 1 drop of a solution containing 1 part of resorcin in 200 parts of water, and pour the mixture cautiously into a test tube containing 3 cc of concentrated sulphuric acid, holding the tube in an inclined position in such a manner that the two liquids shall not mix. Allow it to stand 3 minutes, then sway the tube slowly from side to side in such a manner as to produce a gentle rotary motion of the two layers. Persist in this operation, if necessary, for a minute or more, using a piece of white paper for a background, and producing only a very gradual and partial mixing of the acid and water. Nearly half of the acid should remain as a distinct unmixed layer at the end. When methyl alcohol is present, the shaking causes the separation of more or less voluminous flocks of a very characteristic rose-red color. The appearance of colored zones or flocks of other hues, even when tinged with red, or of a rose-red solution without flocks, should never be considered proof of the presence of methyl alcohol. However, if the flocks are reddish-brown, or if the upper layer has a pronounced red, it is often well to repeat the test. By this method for the removal of acetaldehyde 10 per cent of methyl alcohol may be readily detected, and an experienced operator may detect with certainty a less amount.<sup>d</sup>

## 7.—DETERMINATION OF LEMON OIL.

### (a) BY POLARIZATION.

Polarize the extract without dilution in a 200-mm tube at a temperature of 20° C., using the sugar scale. Divide the reading by 3.2 and, in the absence of other optically active substances, the result will be the percentage of lemon oil by volume.<sup>e</sup>

A small amount of cane sugar is occasionally present, being used to facilitate solu-

<sup>a</sup> Mulliken and Seudder advise against the use of the casein test owing to its extreme delicacy and to the fact that minute amounts of formaldehyde may be produced by the oxidation of grain alcohol. *Amer. Chem. Jour.*, 1900, **24**, 444.

<sup>b</sup> Personal communication from A. G. Woodman.

<sup>c</sup> *Amer. Chem. Jour.*, 1899, **21**, 266.

<sup>d</sup> In the examination of other alcoholic liquids the substances interfering with the resorcin test, together with methods for their removal, may be found by consulting the original article. *Amer. Chem. Jour.*, 1899, **21**, 266.

<sup>e</sup> J. C. Mims finds pure oil of lemon to polarize as high as 35° in solutions of 10 per cent by volume.

tion of the oil. In such case wash the "solid residue" from 10 cc of sample with three portions of 5 cc each of ether to remove waxy and fatty matters; dry and weigh residue of cane sugar, deducting 0.38 from the reading for each 0.1 per cent of sugar so found.

(b) BY PRECIPITATION.

Pipette 20 cc of the extract into a Babcock milk flask; add 1 cc dilute hydrochloric acid (1:1); add 25 to 28 cc of water previously warmed to 60° C.; mix and stand in water at 60° for five minutes; whirl in centrifuge for five minutes; fill with warm water to bring the oil into the graduated neck of the flask; repeat whirling for two minutes; stand in water at 60° for a few minutes and read the per cent of oil by volume. Where the oil of lemon is present in amounts over 2 per cent add to the percentage of oil found 0.4 per cent to correct for the oil retained in solution. Where less than 2 per cent and more than 1 per cent is present, add 0.3 per cent for correction.

Save the precipitated oil for the determination of refraction.

When the extract is made in accordance with the United States Pharmacopœia the results by the two methods just given should agree within 0.2 per cent.

To obtain per cent by weight from per cent by volume, as found by either of the above methods, multiply the volume percentage by .980 and divide the result by the specific gravity of the original extract.

#### 8.—DETERMINATION OF REFRACTION OF PRECIPITATED OIL.

(a) Determine the refractive index of the precipitated oil as directed under Edible Oils and Fats on page 22. Compare with similar precipitated oil from standard extract. Or,

(b) Place a few drops of the oil obtained above in a Zeiss butyro-refractometer at a temperature of 30°. Normal oil when treated under these conditions will have a refraction of 67° to 72° and a dispersion of 2°.

Limonene and most commercial adulterants give a higher reading, with the exception of citronella aldehyde and oil of turpentine.<sup>a</sup>

#### 9.—DETECTION OF COLORING MATTERS.

Follow directions given under coloring materials (p. 111<sup>r</sup>). For Arata's test (see page 112) the "total residue" may be used after the solution in water. Upon the addition of hydrochloric acid in the determination of oil by precipitation, valuable indications as to the color are frequently given. Tartrazin, naphthol yellows, and curcuma retain their color, a pink or red coloration indicates a tropaeolin, while Martius' yellow and salts of di-nitrocresol are precipitated with decolorization of the extract.

### XIII.—FRUITS AND FRUIT PRODUCTS.

By L. S. MUNSON and L. M. TOLMAN,  
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#### 1. GENERAL DISCUSSION.

In the examination of fruits and fruit products, much depends upon the object in view; and the preparation of samples, and the determinations made will depend largely upon the judgment of the analyst. For example, in the determination of heavy metals in canned fruits, one would not be justified in using the liquor, but should work on the pulped contents of the can, while for the detection of glucose,

<sup>a</sup> Jour. Amer. Chem. Soc., 1899, 21, 1132.

preservatives, or coloring matter, examination of the liquor would be sufficient. The relative weights of liquor and fruit may be of value in detecting the presence of an excessive amount of water. A. S. Mitchell<sup>a</sup> has noted the presence of free sulphuric acid in jellies. The methods for determination of mineral acids in vinegar can be readily applied to fruit products. The presence of phosphoric acid would be shown in the examination of the ash.

The determination of solids by drying at 100° C. gives lower results than drying in vacuo at lower temperature, or calculation from the specific gravity of the solution, the reason undoubtedly being that levulose is dehydrated at 100° C.<sup>b</sup> But as few laboratories are equipped to use the drying in vacuo method, and as it is not possible to determine specific gravity in all cases, it is necessary to adopt some method which will give uniform results. In such a method the empirical rules have to be followed closely in order to obtain comparative results.

## 2. PREPARATION OF SAMPLE.

### (a) JUICES, JELLIES, AND SIRUPS.

Prepare the fresh juices by pressing in a jelly bag the well pulped fruit and filtering through muslin. In the case of fresh fruit juices and fresh fruits the determination of total and volatile acids and sugars, and the polarization should be made at once, as fermentation takes place in a very short time. Portions for polarization and reducing sugar may be weighed out and an excess of lead sub-acetate added. They can then be kept for several days, if desired, without fermentation. All samples must be transferred without delay to glass-stoppered bottles and kept in a cool place.

In the case of jellies, thoroughly mix to ensure uniformity in sampling. Weigh 60 grams into a 300-cc flask, dissolve in water by means of frequent shaking, make up to the mark with water, and use aliquot portions for the various determinations. With jellies that contain starch or other insoluble material, thoroughly mix before taking aliquot portions for the various determinations.

Dealcoholize sirups by evaporation to one-third their volume and dilute with water till they contain from 15 to 20 per cent of solids.

### (b) FRESH FRUITS.

Pulp the whole, well-cleaned fruit in a large mortar or by means of a food chopper and mix thoroughly. In case of stone fruits remove the pits and determine their proportion in a weighed sample.

### (c) JAMS, MARMALADES, PRESERVES, AND CANNED FRUITS.

Thoroughly pulp the entire contents of the jar or can, as directed under fresh fruits; with stone fruits remove the pits, and if desired determine their proportion in a weighed sample. In the examination of canned fruits it is often sufficient to merely examine the sirups in which the fruits are preserved. In such cases the liquor may be separated and treated as is prescribed for juices.

## 3.—DETERMINATION OF TOTAL SOLIDS.

### (a) IN JUICES, JELLIES, AND SIRUPS.

(1) *By direct determination.*—Measure 25 cc of a 20 per cent solution [see 2 (a)] of jelly, or weigh 25 grams of juice, into a large flat-bottomed dish which contains

<sup>a</sup> Communicated by letter.

<sup>b</sup> Carr and Sanborn, U. S. Dept. of Agr., Division of Chemistry, Bul. 47, p. 134.

<sup>c</sup> If a pipette be used it must be graduated so as to deliver a definite volume of a 20 per cent sugar solution after draining a definite time.

about 4 or 5 grams of freshly ignited asbestos to absorb it; dry for from 20 to 24 hours in a water-jacketed oven.<sup>a</sup> If care is taken in measuring, this method will be found to be as accurate as weighing. In case of jellies that contain starch or insoluble matter, solids may be determined as directed below under (b).

(2) *By calculation from specific gravity.*—Determine the specific gravity of the solution of jelly or diluted sirup, or of the juice, by means of a Westphal balance, pycnometer, or specific-gravity spindle, and calculate the solids from Table IV.<sup>b</sup>

(b) IN FRESH FRUITS, JAMS, MARMALADES, PRESERVES, AND CANNED GOODS.

Weigh about 20 grams of pulped fresh fruit, or such an amount of fruit products as will give not more than 3 to 4 grams of dried material, into a large flat-bottomed dish containing ignited asbestos; add a few cubic centimeters of water, mix thoroughly, and dry as in [(a) 1].

#### 4.—DETERMINATION OF INSOLUBLE SOLIDS.

(a) KREMLA'S METHOD MODIFIED.

Weigh 50 grams of the sample; transfer by the aid of warm water to a mortar and thoroughly macerate,<sup>c</sup> then transfer to a muslin filter and wash thoroughly with warm water, care being taken at each addition of water to thoroughly stir the pulp. Collect the filtrate in a 500-cc flask, cool and make up to volume. Usually this amount is sufficient to remove all soluble material. In extreme cases increase the washings to 1000 cc; transfer the insoluble residue to an evaporating dish, dry, and weigh.

(b) GERMAN OFFICIAL METHOD.

Transfer a weighed portion of the fruit product to a graduated flask, add water, shake thoroughly and make up to volume. Allow this to settle and either filter or decant off the supernatant liquid. Take an aliquot for the determination of soluble solids. Total solids less soluble solids equals insoluble solids. The fruit must be thoroughly macerated and the use of a mechanical shaker would be advisable.

#### 5.—DETERMINATION OF ALCOHOL.

Determine alcohol in 50 grams of the original material according to the method prescribed on page 82.

#### 6.—DETERMINATION OF ASH.

Evaporate to dryness 50 cc of the solution of jelly or diluted sirup [see 2 (a)], 25 grams of juice or fresh fruit, or 10 grams of jam, marmalade, preserves, or canned fruit in a large platinum dish; then thoroughly char at as low a heat as possible, extract with water, filter, and wash. Return the filter paper and insoluble material to the dish and thoroughly ignite; add the soluble portion and evaporate the whole to dryness after adding a few cubic centimeters of a solution of ammonium carbonate; then heat for a moment to very low redness; cool in a desiccator and weigh. The weighing must be made as quickly as possible, as the ash absorbs moisture very rapidly.

<sup>a</sup> A. McGill, Laboratory of Internal Revenue, Ottawa, Canada, has devised a forced draft water oven for drying at temperatures between 60° and 90° C. The oven is heated by means of ordinary gas burners, and the temperature is controlled by introducing at the bottom of the oven a blast of air from a blower run by a small water motor. Before discharging into the oven the air tube enters the water chamber and is coiled a number of times in order to sufficiently warm the water before it enters the oven. The exit end of the air tube is covered with a concave-convex disc in order to distribute the blast and to prevent harmful currents. By regulating the burners and the flow of air a fairly constant temperature can be obtained. The bottom of the oven is curved instead of flat, to prevent bumping when the water is boiling; a perforated plate serves as a false bottom.

<sup>b</sup> Ztschr. Nahr. Hyg. Waar. 1892, 6, 483.

<sup>c</sup> McGill, by letter, recommends the use of a mechanical shaker to obtain complete solution of the soluble material.

## 7.—EXAMINATION OF ASH.

## (a) ALKALINITY OF THE ASH.

Into the platinum dish containing the ash run an excess of fifth-normal nitric acid and add a few drops of methyl orange. Carefully rub up the ash with a rubber tipped stirring rod and titrate the excess of acid with decinormal potassium hydroxid. Calculate the alkalinity to per cent of potassium carbonate in the original substance. One cubic centimeter of decinormal acid equals 0.00691 gram of potassium carbonate.

## (b) SULPHATES AND CHLORIDS.

Wash the ash into a 50-cc flask and make up to the mark with water. In 25 cc of this solution determine the sulphates by precipitation with barium chlorid. The weight of barium sulphate times 0.7478 gives the weight of sulphates calculated as potassium sulphate.

In the other portion of the solution determine the chlorids by the Volhard<sup>a</sup> method for chlorin. The nitric acid added before making the titration will, if it contain enough nitrous oxid, completely destroy the red color of the methyl orange and leave a clear solution for the titration. Calculate the chlorid as per cent of sodium chlorid. Pure fruit jellies and jams give practically no chlorids or sulphates in this amount of ash, but glucose goods give appreciable amounts. If it is desired to make a complete ash analysis of juices or fresh fruits much larger amounts will have to be ashed.

## 8.—DETERMINATION OF TOTAL ACIDITY.

Take 25 cc of the solution of jelly or diluted sirup [see 2 (a)], 10 grams of juice or fresh fruit, or 50 cc of the washings from the determination of insoluble solids, and dilute with recently boiled distilled water to about 250 cc, or less if the jelly be not highly colored; add phenolphthalein and titrate the acid with decinormal potassium hydroxid. In case of highly colored products litmus paper may be used instead of phenolphthalein. Calculate the results as sulphuric acid.<sup>b</sup>

It is very desirable that acidity be so expressed as to allow of comparison. This end is not attained by expressing the acidity in terms of the dominant acid of the various fruits; hence  $H_2SO_4$  has been suggested, and already a number of laboratories have used this as a basis.

## 9.—DETERMINATION OF VOLATILE ACIDS.

The determination of volatile acids in fruit products may be desirable in cases where fermentation or the use of decayed fruit is suspected. Dissolve 25 grams of substance, dilute to 50 cc, and distill in a current of steam until about 200-cc have passed over. Titrate the distillate with decinormal potassium hydroxid and express the results as acetic acid. Each cubic centimeter of decinormal alkali is equivalent to 0.006 gram of acetic acid.

## 10.—DETECTION OF FREE MINERAL ACIDS.

A. S. Mitchell and A. E. Leach have both called attention to the presence of free sulphuric and phosphoric acid in jellies. For method of detection see Hehner's method, page 64.

## 11.—DETERMINATION OF NITROGEN.

Use 5 grams of jelly or other fruit product or 10 grams of juice or fresh fruit for the determination of nitrogen according to either the Gunning or the Kjeldahl method. Express results as protein (nitrogen multiplied by 6.25).

<sup>a</sup>Ann. d. Chem. 1877, 190, 1. Sutton, Volumetric Analyses, eighth edition, p. 155.

<sup>b</sup>See Composition of Jellies and Jams. Tolman, Munson, and Bigelow. Jour. Am. Chem. Soc. 1901, 23, 348.

## 12.—POLARIZATION.

Dissolve half the normal weight of jelly or other fruit product, or the normal weight of juices or fresh fruits, in a sufficient quantity of water in a 100-cc sugar flask, add an excess of lead subacetate (from 5 to 10 cc, see footnote, p. 84), filter, and polarize in a 200-mm tube, observing the temperature of the solution. Invert 50 cc of this solution using 5 cc of hydrochloric acid and heating to 68° C. in 15 minutes. Polarize in a 220-mm tube at the same temperature as was employed in making the direct reading.

On account of the large amounts of invert sugar usually found in these products it is necessary that the direct and invert readings should be made at the same temperature.

## 13.—DETERMINATION OF CANE SUGAR.

## (a) BY POLARIZATION.

Calculate cane sugar from the direct and the invert readings according to Clerget's formula:

$$S = \frac{100 (a - b)}{144 - \frac{t}{2}}$$

## (b) BY INVERSION.

Where only a small amount of cane sugar is present it is best determined by calculation from the increase in reducing sugars after inversion. For this purpose treat 5 grams of jelly, sirup, or other fruit product, or 25 grams of juice or fresh fruit with lead subacetate in excess, and after making up to 100 cc and filtering invert 50 cc in a 100-cc flask with 5 cc of hydrochloric acid. After inversion neutralize the acid with sodium hydroxid, precipitate excess of lead with sodium sulphate and increase in volume to 100 cc. Filter and dilute so that the solution does not contain more than 1 per cent of reducing sugar. The per cent increase in reducing sugar after inversion multiplied by 0.95 equals per cent of cane sugar.

## 14.—DETERMINATION OF REDUCING SUGARS.

Treat 5 grams of jelly (25 cc of a 20 per cent solution [2 (a)] may be employed), sirup, or other fruit product, or 25 grams of juice or fresh fruit with lead subacetate in excess (2 to 5 cc); make up to 100 cc and filter. Transfer from 25 to 50 cc—depending upon the per cent of reducing sugar present—to a 100-cc flask and add a saturated solution of sodium sulphate in sufficient amount to precipitate the excess of lead; complete the volume to 100 cc and use the filtered solution for the determination of reducing sugars. The approximate amount of reducing sugar present may be readily ascertained from the polarizations and from the percentage of solids. Use Allihn's method for the determination (p. 49).<sup>a</sup>

## 15.—DETERMINATION OF DEXTRIN.

Dissolve 10 grams of the sample<sup>b</sup> in a 100-cc flask; add 20 mg of potassium fluorid and then about one-quarter of a cake of compressed yeast.<sup>c</sup> Allow the fermentation to proceed below 25° C. for 2 or 3 hours to prevent excessive foaming, and then place in an incubator at a temperature of from 27° to 30° C. for 5 days. At the end of that time, clarify with lead subacetate and alumina cream; make up to 100 cc and polarize in a 200-mm tube. A pure fruit jelly will show a rotation of not more than a few

<sup>a</sup> U. S. Department of Agriculture, Division of Chemistry, Bulletin 46 revised, page 35.

<sup>b</sup> In the case of jellies, 50 cc of a 20 per cent solution, prepared as directed under 1 (a), may be used.

<sup>c</sup> Bigelow and McElroy. Jour. Am. Chem Soc. 1893, 15, 668.

tenths of a degree either to the right or to the left.<sup>a</sup> If a Schmidt and Haensch polariscope be used and a 10 per cent solution be polarized in a 200-mm tube, the number of degrees read on the sugar scale of the instrument multiplied by 0.8755 will give the percentage of dextrin, or the following formula<sup>b</sup> may be used:

$$\text{Percentage of dextrin} = \frac{C \times 1,000 \times V}{198 \times L \times W}$$

in which

C=degrees of circular rotation,

V=volume in cubic centimeters of solution polarized,

L=length of tube in centimeters,

W=weight of sample in solution in grams.

#### 16.—DETERMINATION OF ALCOHOL PRECIPITATE.

Take 100 cc of a 20 per cent solution of jelly [see 2 (a)], diluted sirup, or of the washings from the determination of insoluble solids, and evaporate to 20 cc; then add slowly and with constant stirring 200 cc of 95 to 96 per cent alcohol and allow the mixture to stand overnight. Filter and wash with 80 per cent alcohol by volume. Wash this precipitate off the filter paper with hot water into a platinum dish; evaporate to dryness; dry at 100° C. for several hours and weigh; then burn off the organic matter and weigh the residue as ash. The loss in weight upon ignition is called alcohol precipitate.

The ash should be largely lime and not more than 5 per cent of the total weight of the alcohol precipitate. If it is larger than this some of the salts of the organic acids have been brought down. Titrate the water-soluble portion of this ash with decinormal acid, as any potassium bitartrate precipitated by the alcohol can thus be estimated.

The general appearance of the alcohol precipitate is one of the best indications as to the presence of glucose and dextrin. Upon the addition of alcohol to a pure fruit product a flocculent precipitate is formed with no turbidity, while in the presence of glucose a white turbidity appears at once upon adding the alcohol, and a thick, gummy precipitate forms.

#### 17.—DETERMINATION OF TARTARIC, CITRIC, AND MALIC ACIDS.<sup>c</sup>

Use the filtrate from the alcohol precipitate in the determination of the organic acids. After evaporating off the alcohol and taking up the acids with water add lead subacetate until the solution is alkaline, then filter and wash the precipitate until only a slight amount of lead remains in the washings. Wash the precipitate off the filter paper into a beaker with hot water, precipitate the lead by hydrogen sulphid and filter off the lead sulphid while hot, washing with hot water. Evaporate the filtrate which contains the free organic acids to about 50 cc, neutralize exactly with potassium hydroxid, add an excess of strong solution of neutral calcium acetate with constant stirring, and allow to stand from 6 to 12 hours. Throw the precipitate of calcium tartrate on a filter paper and wash until filtrate and washings make exactly 100 cc; ignite the filter paper and precipitate, and determine the lime and tartaric acid by titration. A correction of 0.0286 grams of tartaric acid, which is held in solution in the 100 cc of washings as calcium tartrate, must be added. Now evaporate the filtrate down to about 20 cc, and if a precipitate of calcium citrate is formed filter it off hot, wash with hot water, ignite, and titrate the lime. From

<sup>a</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 66.

<sup>b</sup> Wiley, Chem. News, 1882, 46, 175.

<sup>c</sup> A modification of Schmidt & Hiepe's method. U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 67; Ztschr. anal. Chem., 1882, 21, 534-541.

this calculate the citric acid. Again evaporate the filtrate to about 20 cc and add 3 volumes of 96 per cent alcohol by volume, which will throw down the calcium salt of tartaric acid held in solution, the rest of the citrate, and the malate and succinate. Filter this off, ignite, titrate, and calculate as malic acid, after subtracting the tartaric acid present, as the amount of citric and succinic acid present is very small.

#### 18.—DETERMINATION OF TARTARIC ACID. <sup>a</sup>

To 100 cc of the fruit juice add 2 cc of glacial acetic acid, 2 or 3 drops of a 20 per cent potassium acetate solution and 15 grams of pure finely powdered potassium chlorid, dissolve this by shaking, and then add 20 cc of 96 per cent alcohol. Then stir vigorously for one minute, rubbing the walls of the beaker with the glass stirring rod to start the crystallization of the potassium bitartrate. Allow to stand 15 hours at room temperature. Filter and wash the precipitate onto a Gooch crucible with a thin asbestos felt, using the vacuum pump. Wash with a mixture of 15 grams of potassium chlorid, 20 cc alcohol, and 100 cc water. The beaker is rinsed three times with a few cubic centimeters of this solution. The precipitate is also washed with a few cubic centimeters, but so that not more than 20 cc in all of the wash solution is used. The precipitate and asbestos filter are washed back into the beaker and heated to boiling. While still hot the solution is titrated with decinormal alkali, using phenolphthalein as indicator. To the amount of alkali used must be added 15 cc for the potassium bitartrate remaining dissolved in the solution. One cubic centimeter of decinormal alkali is equivalent to .0150 grams potassium bitartrate.

#### 19.—DETERMINATION OF CITRIC ACID. <sup>b</sup>

Fifty cubic centimeters of the fruit solution is evaporated on the water bath to a sirupy condition. To the residue add, very slowly at first, stirring constantly, 95 per cent alcohol until no further precipitate is formed; 70 to 80 cc are generally enough. Filter and wash the residue with 95 per cent alcohol. Evaporate the filtrate to eliminate the alcohol, take up the residue with a little water and transfer to a graduated cylinder, making up to 10 cc. To 5 cc of this solution add half a cubic centimeter of glacial acetic acid, and to this add, drop by drop, a saturated solution of lead acetate. The presence of citric acid is shown by the appearance of a precipitate which possesses the property of disappearing on being heated and reappearing on cooling. In order to separate the citric acid from other acids, heat to boiling, filter, and wash with boiling water; then allow to cool and the precipitate of lead citrate will re-form. This lead precipitate may be filtered off, washed into weak alcohol, dried, weighed, and the citric acid calculated. It is necessary that there shall be no tartaric acid present. If the tartaric acid has been estimated, any error on this account may be avoided by adding enough decinormal potash to neutralize the tartaric acid before the alcohol is added.

#### 20.—DETECTION OF PRESERVATIVES.

Dissolve about 25 grams of the sample in water, acidify, and extract with ether. Remove the ether layer and allow it to evaporate spontaneously. Take up the residue, which may contain salicylic and benzoic acids and saccharin, with water. For detecting preservatives so separated, and to test further for preservatives, use methods described by the referee on that subject (p. 107).

#### 21.—DETECTION OF COLORING MATTER.

Follow directions given on pages 111 and following.

#### 22.—DETECTION OF ARTIFICIAL SWEETENING MATERIALS.

Follow directions given on page 89.

<sup>a</sup> Halenke & Möslinger, *Ztschr. anal. Chem.*, 1895, **34**, 283.

<sup>b</sup> Möslinger, *Ztschr. Unter. Nahr. u. Genuss.*, 1899, **2**: 93.



## 23.—DETECTION OF STARCH.

First destroy the color of the jelly by treatment with sulphuric acid and potassium permanganate and then test with iodine. Bring the solution of jelly nearly to the point of boiling, add several cubic centimeters of dilute sulphuric acid and then potassium permanganate until all color is destroyed. By this treatment the starch remains unaffected. The test for starch is not necessarily an indication of its addition as an adulterant. It is almost always present in the apple, and occasionally in other fruits, and unless it is present in the jelly or other fruit product in considerable amounts it may be due to that source.

## 24.—DETECTION OF GELATIN.

The presence of gelatin in jellies and jams is shown by a higher content of nitrogen. Precipitate a concentrated solution of jelly or jam with 10 volumes of absolute alcohol and determine nitrogen in dried precipitate by the Gunning method.<sup>a</sup>

25.—DETECTION OF AGAR AGAR.<sup>b</sup>

Cook the jelly with 5 per cent sulphuric acid, add a crystal of potassium permanganate and allow to settle. If agar is present the sediment will be rich in diatoms, which can be detected by use of microscope.

## 26.—THE DETERMINATION OF HEAVY METALS.

Treat 100 grams of the preserve directly in a large porcelain evaporating dish with sufficient concentrated sulphuric acid to thoroughly carbonize the mass. If much water is present evaporate the material to a sirupy consistency before treating with the acid. From 15 to 25 cc of strong acid has been found sufficient to thoroughly carbonize the amount specified. Then ash the material, transfer the ash to a beaker of about 400-cc capacity, slightly acidify it with hydrochloric acid, and boil for a few moments. Methods for separation and determination of metals are given on page 52.

**XIV.—FERMENTED AND DISTILLED LIQUORS.**

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**(A) WINE.**

The determinations of most value in judging the purity of wine are alcohol, glycerol, extract, ash, total and volatile acids, and reducing sugar. The actual percentage of these substances present is of interest, but much more important are certain relations between them, such as ash to extract, extract to alcohol, alcohol to glycerol, alcohol to acids, and volatile acids to total acids. Examination for preservatives and foreign coloring matter must also be made. Search is sometimes made for lead, which may result from cleansing bottles with the aid of shot; for copper and arsenic, which sometimes result from the use of insecticides and fungicides on the grapes; and for barium and strontium, which are sometimes used in southern Europe to remove the excess of sulphate introduced by plastering. A qualitative test is often made for nitrates to detect the addition of (impure) water, and for dextrin to determine whether glucose has been used in the preparation of the wine.

Information regarding the manner of preparing the wine is often afforded by the determination of tannin, potassium sulphate (for plastered wines), and of phosphoric acid.

<sup>a</sup> A. Boemer, Chem. Ztg., 1895, 19, 552.

<sup>b</sup> C. Marpmann. Ztschr. f. angew. Mikrosk. 1896, 2, 260.

For the various determinations, a measured volume can be taken more conveniently than a weighed quantity. The results can be calculated to per cent by weight by dividing the results expressed as grams per 100 cc by the specific gravity.

### 1.—DETERMINATION OF SPECIFIC GRAVITY.

Determine specific gravity at the temperature of 15.6° C. by means of the pycnometer, small accurately graduated hydrometer, Westphal balance, or a Westphal plummet on the analytical balance. The pycnometer, when used, should be heated quickly to room temperature after filling and before weighing, to prevent the error due to the collection of moisture on the outside. A small hole filed in the cap will permit the necessary expansion in the volume of liquid.

### 2.—DETERMINATION OF ALCOHOL.

Measure 100 cc of the liquid into an Erlenmeyer flask of from 250 to 300-cc capacity; add 50 cc of water; attach the flask to a vertical condenser by means of a bent tube and distill 100 cc. Foaming, which sometimes occurs, especially with new wines, may be prevented by the addition of a small amount of tannin. If it be desired to determine alcohol in wines which have undergone acetic fermentation and contain a large amount of acetic acid, 0.1 or 0.2 gram of precipitated calcium carbonate should be added. This is unnecessary, however, in wines of normal taste and odor. Where only occasional determinations of alcohol are made it is found convenient to use an alembic Saleron. This apparatus is made of copper, and it can be readily taken apart and placed in a small box. No rubber connections are necessary, and the setting up requires but a few minutes. Determine the specific gravity of the distillate as directed under Specific Gravity, and obtain the corresponding percentage of alcohol, by volume and grams per 100 cc, from Table II.

### 3.—DETERMINATION OF GLYCEROL.

Evaporate 100 cc of wine<sup>a</sup> in a porcelain dish on the water bath to a volume of about 10 cc and treat the residue with about 5 grams of fine sand and with from 1.5 to 2 cc of milk of lime (containing about 40 per cent of calcium hydroxide or 30 per cent of calcium oxide) for each gram of extract present, and evaporate almost to dryness. Treat the moist residue with 5 cc of 96 per cent alcohol (sp. gr. 0.81), remove the substance adhering to the sides of the dish with a spatula, and rub the whole mass to a paste, with the addition of a little more alcohol. Heat the mixture on the water bath, with constant stirring, to incipient boiling, and decant the liquid into a flask graduated at 100 and 110 cc. Wash the residue repeatedly by decantation with 10 cc portions of hot 96 per cent alcohol. Cool the contents of the flask to 15°, dilute to the 110-cc mark with 96 per cent alcohol, and filter through a folded filter. Evaporate 100 cc of the filtrate to a sirupy consistency in a porcelain dish, on a hot, but not boiling, water bath, transfer the residue to a small glass-stoppered graduated cylinder with 20 cc of absolute alcohol, and add three portions of 10 cc each of absolute ether, mixing after each addition. Let stand until clear, then pour off through a filter, and wash the cylinder and filter with a mixture of one part absolute alcohol to one and one-half parts of absolute ether, pouring the wash liquor also through the filter. Evaporate the filtrate to a sirupy consistency, dry for one hour at the temperature of boiling water, weigh, ignite, and weigh again. The loss on ignition increased by one-tenth gives the glycerol expressed in grams per 100 cc.

<sup>a</sup> With wines whose extract exceeds 5 grams per 100 cc, heat to boiling in a flask the portion to be used in the determination of glycerol, and treat with successive small portions of milk of lime until it becomes, first, darker, and then light in color. When cool, add 200 cc of 96 per cent alcohol (sp. gr. 0.8118), allow the precipitate to subside, filter, and wash with 96 per cent alcohol (sp. gr. 0.8118). Evaporate the filtrate to about 10 cc, add about 5 grams of sand and from 1.5 to 2 cc of milk of lime and proceed as directed above.

## 4.—DETERMINATION OF EXTRACT.

## (a) FROM SPECIFIC GRAVITY OF DEALCOHOLIZED WINE.

Preliminary to its exact determination, the extract should be calculated by the formula:

$$sp=1+x-x'$$

in which  $sp$  is the specific gravity of the dealcoholized wine,  $x$  the specific gravity of the wine,  $x'$  the specific gravity of the alcoholic distillate obtained in the estimation of alcohol.

Illustration.—A sample of Catawba is examined with the result:

Specific gravity of wine ( $x$ ) .....	1.0402
Specific gravity of alcoholic distillate ( $x'$ ) .....	.9857
Difference ( $x-x'$ ) .....	.0545
Specific gravity dealcoholized wine ( $1+x-x'$ ) .....	1.0545
Extract (from Table V) .....	14.48 grams per 100 cc.

The extract content equivalent to  $sp$  is obtained from table.

## (b) BY EVAPORATION.

(1) *In dry wines.*

(Having an extract content of less than 3 grams per 100 cc).

Evaporate 50 cc of the sample on the water bath to a sirupy consistence in a flat-bottom platinum dish about 85 mm in diameter and capable of holding about 75 cc. Heat the residue for two and a half hours in a drying oven at the temperature of boiling water and weigh. This weight multiplied by 2 gives grams of total residue in 100 cc. The sugar-free extract is found by deducting the weight of sugar in excess of 0.1 gram per 100 cc from the total residue. In the case of plastered wines, the potassium sulphate in excess of 0.1 gram is also deducted.

(2) *In sweet wines.*

When the extract content is between 3 and 6 grams per 100 cc treat 25 cc of the sample as described under dry wines. When the extract exceeds 6 grams per 100 cc, however, the result obtained under (a) is accepted, and it is not attempted to determine it gravimetrically. This is because of the serious error connected with drying levulose at high temperature. The table referred to here was obtained by drying at 75° C. in vacuo.

## 5.—DETERMINATION OF ASH.

Ignite at low redness, until thoroughly charred, the residue from the determination of extract,<sup>a</sup> exhaust with water, filter, and wash. Return the filter paper and insoluble material to the dish and burn to a white ash, add the soluble portion and evaporate the whole to dryness, heat to a low redness, cool in a desiccator, and weigh. With dry wines complete combustion can often be obtained without leaching.

## 6.—DETERMINATION OF TOTAL ACIDS.

Expel any carbon dioxid that is present by continued shaking. Transfer 25 cc of the sample to a beaker, heat to incipient boiling, and, in the case of white wines, add about 10 drops of a neutral litmus solution and titrate while still hot with decinormal sodium hydroxid solution. With red wines, add decinormal sodium hydroxid solution until the red color changes to violet, and continue adding a few drops at a time until a drop of the mixture placed on delicate neutral litmus paper ceases to show an acid reaction.<sup>b</sup> The result is expressed in terms of tartaric acid.

<sup>a</sup> Employ the residue obtained by evaporating 25 cc of the wine when the extract has been calculated from specific gravity.

<sup>b</sup> See Appendix, p. 155.

One cubic centimeter of decinormal sodium hydroxid solution is equivalent to 0.0075 gram tartaric acid.

#### 7.—DETERMINATION OF VOLATILE ACIDS.

Distill, in a current of steam, 50 cc of wine, to which a little tannin has been added to prevent foaming. Heat the flask containing the sample until the liquid boils, lower the flame under it and pass the steam through until 200 cc have been collected in the receiver; titrate the distillate with decinormal sodium hydroxid solution, using phenolphthalein as indicator, and express the result as acetic acid.

One cubic centimeter of decinormal sodium hydroxid solution is equivalent to 0.006 gram acetic acid.

#### 8.—DETERMINATION OF FIXED ACIDS.

The amount of fixed acids is ascertained by subtracting 1.25 times the volatile acids from the total acids expressed as tartaric.

#### 9.—DETERMINATION OF UNDETERMINED EXTRACT.

The amount of undetermined extract is ascertained by subtracting the sum of the glycerol, ash, protein, and fixed acids from the weight of the sugar-free extract.

#### 10.—DETERMINATION OF SUGAR.<sup>a</sup>

##### (a) PREPARATION OF SOLUTION.

Place 200 cc of wine in a porcelain dish, exactly neutralize with an approximately normal solution of sodium hydroxid, using litmus paper as indicator, and evaporate to about one-fourth the original volume. Transfer to a 200-cc flask, add sufficient basic lead acetate<sup>b</sup> to clarify, dilute to the mark with water, shake, and filter through a ribbed filter. Transfer 100 cc of the filtrate to a flask graduated at 100 and 110 cc, fill to the upper mark with a saturated solution of sodium sulphate, shake and filter.

##### (b) POLARIZATION.

##### (1) *Direct.*

Polarize part of the filtrate in a 200-mm tube, in a Schmidt and Haensch polariscope, and increase the reading by one-tenth for the polariscope reading. In case the reading is taken on some other instrument than the Schmidt and Haensch it may be calculated by the following data:

1° Ventzke	=0.3468° angular rotation D.
1° angular rotation D	=2.8835° Ventzke.
1° Ventzke	=2.6048° Wild (sugar scale).
1° Wild (sugar scale)	=0.3840° Ventzke.
1° Wild (sugar scale)	=0.1331° angular rotation D.
1° angular rotation D	=0.7511° Wild (sugar scale).
1° Laurent (sugar scale)	=0.2167° angular rotation D.
1° angular rotation D	=4.6154° Laurent (sugar scale).

##### (2) *Invert.*

In order to determine the presence or absence of sucrose it is necessary to subject the sugars to inversion. The filtrate from the lead sulphate obtained in (a) may be

<sup>a</sup> See Appendix, p. 156.

<sup>b</sup> Prepared by boiling for half an hour 430 grams of normal lead acetate, 130 grams of litharge, and 1000 cc of water. The mixture is allowed to cool and settle. when the supernatant liquid is diluted to 1.25 specific gravity with recently boiled water

conveniently employed for this purpose. Fill a flask graduated at 50 and 55 cc to the 50-cc mark with the filtrate, add 5 cc of concentrated hydrochloric acid, invert; polarize in a 220-mm tube, and increase the reading one-tenth to allow for dilution.

(3) *After fermentation.*

In the case of wines polarizing between  $+2.3^{\circ}$  and  $+0.9^{\circ}$  the use of glucose in their preparation can be proved or disproved after fermentation by the presence or absence of certain unfermentable constituents.

Dealcoholize 200 cc of wine by evaporating to about one-fourth its volume, and add enough water to the residue to make its sugar content less than 15 per cent. For the purpose of this operation the sugar content of the wine may be assumed to be 2 per cent less than the extract. Add 2 or 3 grams of compressed yeast, let stand at about  $25^{\circ}$  C. for four or five days, when fermentation will be complete.

Evaporate the fermented liquid in a porcelain dish to a thin sirup after the addition of a little sand and a few drops of a 20 per cent solution of potassium acetate. To the residue add 200 cc of 90 per cent alcohol with constant stirring. Separate the alcoholic solution by filtration and evaporate until about 5 cc remain. Mix the residue with washed boneblack, filter into a graduated cylinder, and wash until the filtrate (cooled to  $15^{\circ}$  C.) amounts to 30 cc. When the filtrate shows a dextrorotation of more than  $1.5^{\circ}$  it indicates the presence of the unfermentable constituents of commercial glucose. Results by this method are not reliable with wines that are heavily preserved.

(c) REDUCING SUGARS.

Dilute a portion of the solution prepared as directed under (a) until it does not contain more than 1 per cent of sugar. In making this dilution the sugar-free extract of a wine may be taken as 2 per cent. The number of volumes of water to be added to the filtrate is thus determined by deducting 2 from the total extract. If the wine is not to be polarized, or for any reason a separate portion is to be prepared for the reduction, the dilution may be conveniently made prior to the clarification. Use Allihn's method, expressing the results as dextrose (see p. 49).

(d) CANE SUGAR.

(1) *By reduction.*

Invert a portion of the filtrate obtained in (a) as directed under (b) (2); determine reducing sugars according to (c); deduct from the figure thus obtained the reducing sugars originally present, and multiply the result by 0.95 for conversion into cane sugar.

(2) *By polarization.*

Calculate from the direct and invert polarizations by the Clerget formula:

$$\text{Per cent sucrose} = \frac{100(R - R')}{144 - \frac{T}{2}}$$

(e) COMMERCIAL GLUCOSE. <sup>a</sup>

(1) Wine with not more than 0.1 per cent of reducing sugar, and which polarizes to the left or not more than  $0.9^{\circ}$  to the right, has not been treated with glucose.

(2) Wine with not more than 0.1 per cent of reducing sugar, and which polarizes  $0.9^{\circ}$  or more to the right, may contain dextrin and the unfermentable constituents of commercial glucose. In such a case, examine according to 10 (3) and 11.

(3) If the reducing sugar exceeds 0.1 per cent, examine according to 10 (3) for the unfermentable constituents of commercial glucose.

## 11.—DETERMINATION OF GUM AND DEXTRIN.

Evaporate 100 cc of wine to about 10 cc and add 10 cc of 96 per cent alcohol (sp. gr. 0.81). If gum or dextrin be present (indicated by the formation of a voluminous precipitate), continue the addition of alcohol slowly and with stirring until 100 cc have been added. Let stand over night, filter, and wash with 80 per cent alcohol by volume (sp. gr. 0.84). The precipitate may then be dried and weighed, or it may be treated according to Sachsse's method for the determination of starch.

12.—DETERMINATION OF TANNIN AND COLORING MATTER.<sup>a</sup>

Dealcoholize 100 cc by evaporation and dilute with water to the original volume. Transfer 10 cc to a porcelain dish of about 2 liters capacity; add about a liter of water and exactly 20 cc of indigo<sup>b</sup> solution, measuring the latter by means of a burette. Add decinormal potassium permanganate solution, which has been standardized against decinormal oxalic acid, a cubic centimeter at a time, until the blue color changes to green; then a few drops at a time until the color becomes bright yellow. Designate the number of cubic centimeters of permanganate solution employed by (a).

Treat 10 cc of the dealcoholized wine, prepared as above, with carefully purified boneblack for fifteen minutes; filter and wash the boneblack thoroughly with water. Add a liter of water and 20 cc of indigo solution and titrate with permanganate as above. Designate the number of cubic centimeters of permanganate solution employed by (b).

Then  $a - b = c$  = the number of cubic centimeters of permanganate solution required for the oxidation of the tannin and coloring matter in 10 cc of wine.

Multiply (c) (corrected to cubic centimeters of decinormal solution, if the solution employed is not exactly decinormal) by 0.04157 for tannin and coloring matter, expressed in grams per 100 cc.

One cubic centimeter of decinormal permanganate solution is equivalent to 0.004157 gram tannin.

## 13.—DETERMINATION OF SODIUM CHLORID.

Sodium chlorid is obtained by dissolving the ash in water, slightly acidifying with nitric acid, neutralizing with calcium carbonate, and titrating with silver nitrate, using normal potassium chromate as indicator.

## 14.—DETERMINATION OF POTASSIUM SULPHATE.

Precipitate sulphuric acid directly in 50 cc of wine by means of barium chlorid, and determine the resulting barium sulphate by the ordinary method. Express the result in grams of potassium sulphate per 100 cc. In all cases this determination should be made in the original wine, as results obtained with the ash are always low.

## 15.—DETERMINATION OF PHOSPHORIC ACID.

Determine phosphoric acid in the ash by the official volumetric method. In case the ash has been used for other determinations, and it is necessary to begin with the original wine, evaporate 100 cc of dry wines and ignite directly. With sweet wines, evaporate 100 cc to a sirupy consistency in a flask of about 250-cc capacity, add 25 cc of concentrated sulphuric acid and heat with a low flame till the evolution of gas

<sup>a</sup> Neubauer-Löwenthal method. *Annalen der Oenologie*, 2, 1.

<sup>b</sup> Instead of the indigo-carmin called for by the original method, sodium sulphindigotate may be employed, as suggested by Schroeder (*Ztschr. anal. Chem.*, 1886, 25, 112). To prepare the solution, dissolve 6 grams of sodium sulphindigotate in 500 cc of water with aid of heat; cool; add 50 cc of concentrated sulphuric acid; dilute to 1 liter and filter. (U. S. Dept. of Agr., Bul. 46 revised, p. 66.)

ceases. Add about 75 cc concentrated nitric acid, warm gently, and finally evaporate almost to dryness. Then add 10 cc of concentrated sulphuric acid and a little mercury and boil till the solution clears.<sup>a</sup> Employ the official volumetric method for phosphoric acid, as stated above.

#### 16.—DETERMINATION OF TARTARIC ACID AND TARTRATES.

##### (a) TOTAL TARTARIC ACID.<sup>b</sup>

To 100 cc of wine add 2 cc of glacial acetic acid, 3 drops of a 20 per cent solution of potassium acetate, and 15 grams of powdered potassium chlorid, and stir to hasten solution. Add 15 cc of 95 per cent alcohol (sp. gr. 0.81) and rub the side of the beaker vigorously with a glass rod for about 1 minute to start crystallization. Let stand at least 15 hours at room temperature; decant the liquid from the separated acid potassium tartrate as rapidly as possible (using vacuum) through a Gooch crucible prepared with a very thin film of asbestos, transferring no more of the precipitate to the crucible than necessary. Wash the precipitate and filter three times with a small amount of a mixture of 15 grams potassium chlorid, 20 cc of 95 per cent alcohol (sp. gr. 0.81), and 100 cc water, using not more than 20 cc of the wash solution in all. Transfer the asbestos film and precipitate to the beaker in which the precipitation took place, wash out the Gooch crucible with hot water, add about 50 cc of hot water, heat to boiling, and titrate the hot solution with decinormal sodium hydroxid, using delicate litmus tincture or litmus paper as indicator. Increase the number of cubic centimeters of decinormal alkali employed by 1.5 on account of the solubility of the precipitate. This figure multiplied by 0.015 gives the amount of total tartaric acid in grams per 100 cc.

##### (b) CREAM OF TARTAR.

Ignite the residue obtained from the evaporation of 50 cc of wine as directed under the determination of ash. Exhaust the ash with hot water, add to the filtrate 25 cc of decinormal hydrochloric acid, heat to incipient boiling and titrate with decinormal alkali solution, using litmus as indicator. Deduct from 25 cc the number of cubic centimeters of decinormal alkali employed and multiply the remainder by 0.0188 for potassium bitartrate expressed in grams.

##### (c) FREE TARTARIC ACID.

Add 25 cc of decinormal hydrochloric acid to the ash of 50 cc of wine, heat to incipient boiling and titrate with decinormal sodium hydroxid, using litmus as indicator. Deduct the number of cubic centimeters of alkali employed from 25 and multiply the remainder by 0.0075 to obtain the amount of tartaric acid necessary to combine with all the ash (considering it to consist entirely of potash). Deduct the figure so obtained from the total tartaric acid for the free tartaric acid.

#### 17.—DETERMINATION OF PROTEIN.

Determine nitrogen in 50 cc of wine by the Kjeldahl or the Gunning method, and multiply the result so obtained by 6.25.

#### 18.—DETERMINATION OF HEAVY METALS.

Lead is often found in wine as a result of the use of shot in cleaning bottles, and copper and arsenic may occur in wine made from grapes sprayed with insecticides. Lead and copper may be determined in 500 or 1,000 cc by the method given under Vegetables.

<sup>a</sup> Glaeser and Mühle, Chem. Ztg., 1896, 20, 723.

<sup>b</sup> Halenke and Möslinger, Ztschr. anal. Chem., 1895, 34, 263.

Arsenic may be detected or determined by the Marsh apparatus if combustion be effected by the method given under the determination of phosphoric acid (p. 86).

Copper may be precipitated electrolytically<sup>a</sup> in 500 cc of the undiluted wine by using as electrodes pieces of platinum foil 3 by 15 cm.

#### 19.—DETERMINATION OF BARIUM AND STRONTIUM.<sup>b</sup>

Evaporate to dryness 100 cc of wine, incinerate as directed under the determination of ash (p. 83), dissolve in dilute hydrochloric acid, evaporate to dryness, and examine the residue spectroscopically. If barium or strontium be present, fuse with sodium carbonate<sup>c</sup> to decompose silicates, dissolve in water and determine by precipitation with sulphuric acid.

#### 20.—DETECTION OF FOREIGN COLORING MATTER.

Follow directions given under Coloring Matter (pp. 111 and following).

#### 21.—DETECTION OF NITRATES.

##### (a) WHITE WINE.

Treat a few drops of the wine in a porcelain dish with 2 or 3 cc of concentrated sulphuric acid which contains about 0.1 gram of diphenylamin<sup>d</sup> per 100 cc. The deep blue color formed in the presence of nitrates appears so quickly that it is not obscured, even in sweet wine, by the blackening produced by the action of sulphuric acid on the sugar.

##### (b) RED WINE.

Clarify with basic lead acetate and remove the excess of lead with sodium sulphate, as directed under the determination of sugar (p. 84). Filter, and treat a few drops of the filtrate as directed under (a).

#### 22.—DETECTION OF PRESERVATIVES.

The preservatives to be tested for in wines are salicylic acid, benzoic acid, saccharin, abristol, hydronaphthol, boric acid, borofluorids, and silicofluorids. Of these the salicylic and benzoic acids are both somewhat commonly employed. Abristol is said to be used to some extent in Europe, but has not yet been reported in American wines. Hydronaphthol has been used in rare instances, and is still used with sufficient frequency to warrant more consideration than it usually receives from food laboratories. Boric acid is better known as a preservative for milk and meat preparations than for fruit and fruit preparations. It is sometimes used, however, in both wine and beer. Its detection is a somewhat more complicated matter than is the case with the other preservatives, because a small amount of boric acid is normal to wines. It is sometimes a difficult matter to fix the amount which may naturally occur. In order to make this test of practical value, therefore, it is essential that the determination of boric acid should be quantitative. The alkaline fluorids, as well as the alkaline borofluorids and silicofluorids, are coming into somewhat general use now as food preservatives, although they have not been frequently reported in wines.

<sup>a</sup>Fruhauf and Ursic, Bericht u. die Versammlung Oesterreichischer Oenomiker in Bozen, 1886, p. 66; Borgmann, Anal. des Weines, 2d ed., p. 146.

<sup>b</sup>Borgmann, Anal. des Weines, 2d ed., p. 143.

<sup>c</sup>R. Fresenius, Ztschr. anal. Chem., 1890, **29**, 20, 143 and 413; 1891, **30**, 18, 452 and 583; 1893, **32**, 189 and 312.

<sup>d</sup>Egger, Arch. Hyg., **2**, 373.



## (a) SALICYLIC ACID.

Treat about 75 cc of wine with sufficient basic lead acetate<sup>a</sup> to clarify, and filter through a ribbed filter paper. Add from 5 to 10 cc of dilute sulphuric acid (1-3), allow the precipitated lead sulphate to subside, and decant about 50 cc of the supernatant liquid into a separatory funnel. Extract with ether or chloroform and test for salicylic acid as directed under Food preservatives (p. 108). The writer has obtained much more satisfactory results by extracting after clarification as directed above, than by extracting the wine directly with a mixture of ether and petroleum ether, or by extracting the evaporated residue from the ether extract with petroleum ether. In no case should the volume of wine extracted for the detection of salicylic acid greatly exceed 50 cc. A similar reaction (with ferric chlorid) is said to be obtained sometimes from wines which contain no salicylic acid when a large volume of the wine is employed.<sup>b</sup>

## (b) BENZOIC ACID.

Acidify about 100 cc of wine with dilute (1-3) sulphuric acid, extract with ether and detect by Mohler's method, as described under Food preservatives (p. 109).

The presence of benzoic acid may be confirmed by neutralizing the aqueous solution of the extracted benzoic acid with sodium hydroxid, evaporating to a very small volume, and acidifying with sulphuric acid, when the presence of a large amount of benzoic acid is indicated by the formation of a white flocculent precipitate. The concentrated solution of the sodium salt may be further tested by adding a few drops of phenolphthalein solution, and then a very dilute solution of sodium hydroxid drop by drop, till an alkaline reaction is obtained, and a drop of a 0.5 per cent ferric chlorid solution, which should decolorize the phenolphthalein, when ferric benzoate is precipitated. The appearance of ferric benzoate is markedly different from that of ferric hydroxid, in that it is almost white when viewed by transmitted light and brown by reflected light, whereas ferric hydroxid has a brown color in both cases.

## (c) DETECTION OF SACCHARIN.

Proceed as directed on page 109.

## (d) SUCROL OR DULCIN.

(1) *Morpurgo's method.*<sup>c</sup>

Evaporate about 100 cc of wine to a sirupy consistency after the addition of about 5 grams of lead carbonate, and extract the residue several times with alcohol of about 90 per cent; evaporate the alcoholic extract to dryness; extract the residue with ether, and allow the ether to evaporate spontaneously in a porcelain dish. Now add 2 or 3 drops each of phenol and concentrated sulphuric acid and heat for about five minutes on the water bath; cool; transfer to a test tube and pour ammonia or sodium hydroxid over the surface with the least possible mixing. The presence of dulcin is indicated by the formation of a blue zone at the plane of contact.

(2) *Jorisson's method.*<sup>d</sup>

Suspend the residue from the ether extract obtained as directed above in about 5 cc of water; add from 2 to 4 cc of an approximately 10 per cent solution of mercuric nitrate, and heat from 5 to 10 minutes on the water bath. In the presence of sucrol, a violet blue color is formed, which is changed to a deep violet by the addition of lead peroxid.

<sup>a</sup> See footnote on page 84.

<sup>b</sup> Medicus, *Ztschr. anal. Chem.*, 1896, **35**, 398.

<sup>c</sup> *Ztschr. anal. Chem.*, 1896, **35**, 104.

<sup>d</sup> *ib.*, 628.

## (e) DETERMINATION OF TOTAL SULPHUROUS ACID.

Distill 100 cc of wine in a current of carbon dioxid, after the addition of about 5 cc of a 20 per cent solution of glacial phosphoric acid, until 50 cc have passed over. Collect the distillate in a decinormal iodine solution in a flask closed with a stopper perforated with two holes, through one of which the end of the condenser passes and through the other a U-tube containing a portion of the standardized iodine solution. Twenty-five cc of decinormal iodine solution may be employed, diluted with water to give the desired volume. The method and apparatus may be simplified without material loss in accuracy by omitting the current of carbon dioxid, adding 10 cc of phosphoric acid instead of 5 cc, and dropping into the distilling flask a piece of sodium bicarbonate weighing not more than a gram immediately before attaching to the condenser. The carbon dioxid liberated is not sufficient to expel the air entirely from the apparatus, but will prevent oxidation to a large extent. The U-tube trap may also be omitted if the end of the condenser tube be made to extend below the surface of the iodine solution, and the distillation conducted with a steady flame. When the distillation is finished, wash the contents of the U-tube into the flask and determine the excess of iodine with standardized thiosulphate solution. On account of its lack of permanence, the iodine solution employed should be titrated from time to time with a decinormal thiosulphate solution (containing 24.8 grams  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  per liter). The number of cubic centimeters of decinormal iodine solution employed, less the number of cubic centimeters of thiosulphate solution required at the end of the determination, is multiplied by 0.0032 for the grams of sulphur dioxid per 100 cc of wine.

Fairly accurate results may also be obtained by the following method:

Place 25 cc of a solution of potassium hydroxid containing 56 grams per liter in a flask of approximately 200-cc capacity. Introduce 50 cc of wine by means of a pipette, mix with the potassium hydroxid, and allow the mixture to stand for fifteen minutes with occasional agitation. Add 10 cc of 1-3 sulphuric acid and a few cubic centimeters of starch solution, and titrate the mixture with a N/50 iodine solution. Introduce the iodine solution as rapidly as possible and continue the addition until the blue color will last for several minutes. One cubic centimeter of N/50 iodine solution is equivalent to 0.00064 gram of sulphur dioxid. The number of cubic centimeters of the iodine solution employed, multiplied by 0.00128, gives the weight of the total sulphur dioxid expressed in grams per 100 cc.

## (f) DETERMINATION OF FREE SULPHUROUS ACID.

Treat 50 cc of wine in a flask, having a capacity of approximately 200 cc, with about 5 cc of 1-3 sulphuric acid, add a small piece of sodium carbonate (about 0.5 gram) to expel the air, and titrate the sulphurous acid with N/50 iodine solution, as directed under total sulphurous acid.

One cubic centimeter of N/50 iodine solution is equivalent to 0.00064 gram of sulphur dioxid.

The number of cubic centimeters of iodine solution employed, multiplied by 0.00128, gives the weight of the free sulphurous acid expressed as sulphur dioxid in grams per 100 cc.

## (g) DETECTION OF BETA-NAPHTHOL.

Extract 200 cc of wine with 10 cc of chloroform in a separatory funnel, add a few drops of alcoholic potash to the chloroform extract in a test tube, and place in a boiling water bath for two minutes. The presence of beta-naphthol is indicated by the formation of a deep blue color, which changes through green to yellow.

## (h) DETECTION OF ABRASTOL.

(1) *Sinabaldi's method.*<sup>a</sup>

Make 50 cc of the sample alkaline with a few drops of ammonia and extract with 10 cc of amyl alcohol (ethyl alcohol is added if an emulsion be formed). Decant the amyl alcohol, filter if turbid, and evaporate to dryness. Add to the residue 2 cc of a mixture of equal parts of strong nitric acid and water, heat on the water bath until half of the water is evaporated, and transfer to a test tube with the addition of 1 cc of water. Add about 0.2 gram of ferrous sulphate and an excess of ammonia, drop by drop, with constant shaking. If the resultant precipitate be of a reddish color, dissolve it in a few drops of sulphuric acid, and add ferrous sulphate and ammonia as before. As soon as a dark-colored or greenish precipitate has been obtained, introduce 5 cc of alcohol, dissolve the precipitate in sulphuric acid, and shake the fluid well and filter. In the absence of abrastol this method gives a colorless or light-yellow liquid, while a red color is produced in the presence of 0.01 gram of abrastol.

(2) *Sanglé-Ferrière's method.*<sup>b</sup>

Boil 200 cc of wine with 8 cc of concentrated hydrochloric acid for one hour in a flask with reflux condenser attached. Abrastol is thus converted into beta-naphthol and is detected as directed under (g).

## (i) BORIC ACID.

Boric acid is a normal constituent of wine and its qualitative detection in wine is therefore of little value unless a very heavy reaction is obtained. For methods of detection and estimation, see page 110.

## (j) DETECTION OF FLUORIDS.

(1) *First method.*<sup>c</sup>

Heat to boiling about 100 grams of wine, made slightly alkaline with ammonium carbonate and precipitate the fluorin with 2 or 3 cc of an approximately 10 per cent solution of calcium chlorid. Continue the boiling for five minutes, separate the precipitate by filtration, wash with a little water, dry, and ignite in a platinum crucible. Add 1 cc of strong sulphuric acid, cover the crucible with a watch glass coated with paraffin or wax, with a character marked through the wax so as to permit the watch glass to be etched at some point, and heat on a water bath for an hour at a temperature of from 75° to 80° C. One milligram can be readily detected by this method. The delicacy of the method is impaired by the presence of a small amount of silica in the ash of the wine.

(2) *Second method.*

If it is desired, the preceding method may be varied by mixing a small amount of precipitated silica with the precipitated calcium fluorid and placing it in a crucible covered by a watch glass which is not coated with paraffin, and to which a drop of water is suspended on the underside. Add 1 cc of concentrated sulphuric acid to the crucible, and heat for an hour at the temperature of 70° or 80° C. The silicon fluorid which is formed is decomposed by the water, leaving a gelatinous deposit of silica, while a ring is frequently etched at the circumference of the drop of water. Any fluosilicates and fluoborates present will also be indicated by this reaction.

<sup>a</sup> Mon. Sci., 1893 [4], 7, 842.

<sup>b</sup> Nevire and Hubert, Mon. Sci., 1895 [4], 9, 324.

<sup>c</sup> Comp. rend. 1893, 117, 93.

## (k) DETECTION OF FLUOBORATES AND FLUOSILICATES.

Make about 200 cc of wine alkaline with limewater, evaporate to dryness, and incinerate. Extract the crude ash first obtained with water, to which sufficient acetic acid has been added to decompose carbonates, filter, burn the insoluble portion, extract with dilute acetic acid, and again filter. The insoluble portion now contains calcium silicate and fluorid, while the filtrate will contain all the boric acid present.

(1) *First method.*<sup>a</sup>

Incinerate the filter containing the insoluble portion, mix with a little precipitated silica, and place, with the addition of 1 or 2 cc of concentrated sulphuric acid, in a short test tube which is attached to a small U-tube containing a few drops of water. The test tube is now placed in a beaker of water, which is kept hot on the steam bath for from 30 to 40 minutes. If any fluorid be present the silicon fluorid generated will be decomposed by the water in the U-tube and will form a gelatinous deposit on the walls of the tube.

The filtrate is now tested as directed under boric acid. If both hydrofluoric and boric acids be present, it is probable that they were combined as borofluorid. If, however, silicon fluorid be detected and not boric acid, the operation is repeated without the introduction of the silica, in which case the formation of the silicon skeleton is conclusive of the presence of fluosilicate.<sup>b</sup>

(2) *Second method.*

Incinerate the filter containing the insoluble portion in a platinum crucible, mix with a little precipitated silica, and add 1 cc of concentrated sulphuric acid. Cover the crucible with a watch glass to whose underside a drop of water is suspended, and heat an hour at the temperature of 70° or 80° C. The silicon fluorid which is formed is decomposed by the water, leaving a gelatinous deposit of silica. Test the filtrate for boric acid as described above.

**(B) BEER.**

## 1.—PREPARATION OF SAMPLE.

Transfer the contents of bottle or bottles into a large flask and shake vigorously to hasten the escape of carbon dioxide. The beer may then be poured into a second receptacle from under the foam.

## 2.—DETERMINATION OF SPECIFIC GRAVITY.

Follow the directions given for the determination of specific gravity in wine (p. 82).

## 3.—DETERMINATION OF ALCOHOL.

Follow the directions given for the determination of alcohol in wine (p. 82).

## 4.—DETERMINATION OF EXTRACT.

Ascertain the extract content corresponding to the specific gravity of the dealcoholized beer according to Table III.

For this purpose employ the formula:

$$sp = g + (1 - a)$$

<sup>a</sup> Nevier and Hubert, Mon. Sci., 1895 [4], 9, 324.

<sup>b</sup> It must be remembered that in an ash that contains an appreciable amount of silica, sulphuric acid will liberate silicon fluorid rather than hydrofluoric acid. The presence of a fluosilicate is indicated, therefore, and not of a fluorid.

in which  $sp$  is the specific gravity of the dealcoholized beer,  $g$  the specific gravity of the beer, and  $a$  the specific gravity of the distillate obtained in the determination of alcohol. In place of this formula, the residue from the distillation of alcohol is sometimes diluted to the original volume, and its specific gravity taken. This is often impracticable owing to the necessity of employing tannin to prevent foaming in the distilling flask, and owing to the coagulation of proteids during the distillation.

The extract of beer can not be accurately determined by evaporation and drying at the boiling point of water because of the dehydration of the maltose.

##### 5.—DETERMINATION OF ORIGINAL GRAVITY OF WORT.

The various methods employed to obtain this figure depend on the fact that the sugars yield about half their weight of alcohol when fermented.

Employ the formula:

$$G = sp + si$$

in which  $G$  is the specific gravity of the original wort,  $sp$  the specific gravity of the dealcoholized beer (see 4), and  $si$  the amount of saccharine matter destroyed by fermentation—obtained from the following table:

*Saccharine matter lost by fermentation.*<sup>a</sup>

1—a	0	1	2	3	4	5	6	7	8	9
0.000	.....	0.0003	0.0006	0.0009	0.0012	0.0015	0.0018	0.0021	0.0024	0.0027
.001	0.0030	.0033	.0037	.0041	.0044	.0048	.0051	.0055	.0059	.0062
.002	.0066	.0070	.0074	.0078	.0082	.0086	.0090	.0094	.0098	.0102
.003	.0107	.0111	.0115	.0120	.0124	.0129	.0133	.0138	.0142	.0147
.004	.0151	.0155	.0160	.0164	.0168	.0173	.0177	.0182	.0186	.0191
.005	.0195	.0199	.0204	.0209	.0213	.0218	.0222	.0227	.0231	.0236
.006	.0241	.0245	.0250	.0255	.0260	.0264	.0269	.0274	.0278	.0283
.007	.0288	.0292	.0297	.0302	.0307	.0312	.0317	.0322	.0327	.0332
.008	.0337	.0343	.0348	.0354	.0359	.0365	.0370	.0375	.0380	.0386
.009	.0391	.0397	.0402	.0407	.0412	.0417	.0422	.0427	.0432	.0437
.010	.0442	.0447	.0451	.0456	.0460	.0465	.0476	.0475	.0480	.0485
.011	.0490	.0496	.0501	.0506	.0512	.0517	.0522	.0527	.0533	.0538
.012	.0543	.0549	.0554	.0559	.0564	.0569	.0574	.0579	.0584	.0589
.013	.0594	.0600	.0605	.0611	.0616	.0622	.0627	.0633	.0638	.0643
.014	.0648	.0654	.0659	.0665	.0671	.0676	.0682	.0687	.0693	.0699
.015	.0705	.0711	.0717	.0723	.0729	.0735	.0741	.0747	.0753	.0759

In this table,  $1-a$  is found by deducting from 1.0 the specific gravity of the alcohol distillate obtained in the determination of alcohol. In case of beer of high acidity, it must be increased by the value of  $b$  in the formula

$$b = 0.9 l - 0.14,$$

in which  $l$  is the percentage of acid calculated to lactic acid. The figure 0.14 is taken as the alcoholic equivalent of the average acid content of beer (ordinarily about 0.15 per cent lactic acid), and is deducted for that reason. The table here given is that of Graham, Hofmann, and Redwood,<sup>b</sup> except that it is expressed here as

<sup>a</sup> Allen, Com. Org. Anal., Vol. I.

<sup>b</sup> Report on Original Gravities, 1852; Allen, Commercial Organic Analysis, 3d edition, Vol. I, p. 136.

specific gravity instead of parts per thousand. It has been adopted by the English excise.

The method outlined above was employed by the same authors, except that they determined the specific gravity of the dealcoholized beer, directly on the residue from the alcohol determination after diluting to the volume of beer employed.

#### 6.—DETERMINATION OF THE DEGREE OF FERMENTATION.

Calculate from the formula

$$D = \frac{100 \text{ } sp}{G}$$

in which  $D$  is the degree of fermentation,  $sp$  the specific gravity of the dealcoholized beer, and  $G$  the gravity of the original wort.

#### 7.—DETERMINATION OF TOTAL ACIDS.

Heat 20 cc of the sample to incipient boiling to liberate carbon dioxid, and titrate with decinormal sodium hydroxid, using neutral litmus paper as indicator. Each cubic centimeter of decinormal alkali employed is equivalent to 0.009 grams of lactic acid. The number of cubic centimeters of decinormal alkali employed in titrating 20 cc of beer is multiplied by 0.045 for the acidity expressed as grams of lactic acid per 100 cc.

#### 8.—DETERMINATION OF VOLATILE ACIDS.

Follow the directions given for the determination of volatile acids in wine (p. 84). This determination is rarely of value in sound beer.

#### 9.—DETERMINATION OF REDUCING SUGAR.

Proceed as directed on page 85, but boil four minutes instead of two. Express the result in terms of maltose equivalent to copper reduced, according to Table IX.

#### 10.—DETERMINATION OF DEXTRIN.

Deduct from the extract the sum of the maltose, protein, glycerol, total acids, and ash. While this method is only approximate, it is sufficiently accurate for most purposes.

If a more accurate determination is desired, 50 cc may be treated by Sachsse's method for the hydrolization of starch, and dextrose determined by copper reduction, according to Allihn's method. From the amount of dextrose so found, 95 per cent of the amount of maltose present in the beer is deducted (20 parts maltose are equivalent to 19 parts dextrose) and the remainder multiplied by 0.9.

#### 11.—DETERMINATION OF GLYCEROL.

Proceed as directed on page 82. The milk of lime is added during evaporation after the carbon dioxid has been expelled.

#### 12.—DETERMINATION OF ASH.

Evaporate 25 cc to dryness, ignite as directed on page 83, and weigh.

#### 13.—DETERMINATION OF PHOSPHORIC ACID.

Employ the official gravimetric or volumetric method,\* using the residue obtained in the determination of ash.

#### 14.—DETERMINATION OF PROTEIN.

Employ the Kjeldahl or the Gunning method for the determination of nitrogen, and multiply the result by 6.25.

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\* U. S. Dept. of Agr., Div. of Chem., Bul. 46, p. 13.

## 15.—DETERMINATION OF CARBON DIOXID.

## (a) BOTTLED GOODS.

Pierce the cork with a champagne tap.<sup>a</sup> Connect with a suitable absorption apparatus, placing an Erlenmeyer flask between the bottle and absorption tubes to allow the bubbles to break and prevent them from passing beyond it. The accompanying illustration (fig. 3) of an apparatus devised by Crampton and Trescot<sup>b</sup> answers admirably for this purpose. Immerse the bottle in water in a suitable vessel—such as an ether can with the top cut away, as shown in the cut—allow the gas to escape slowly, and when it ceases to flow spontaneously heat gradually to about 80° C. and maintain this temperature for about half an hour, shaking the bottle from time to time. Then disconnect the bottle, replace it with a soda-lime tube and draw a current of air through the apparatus. The increase in weight of the absorption tube gives the amount of carbon dioxide. The volume of beer employed is also weighed or measured.

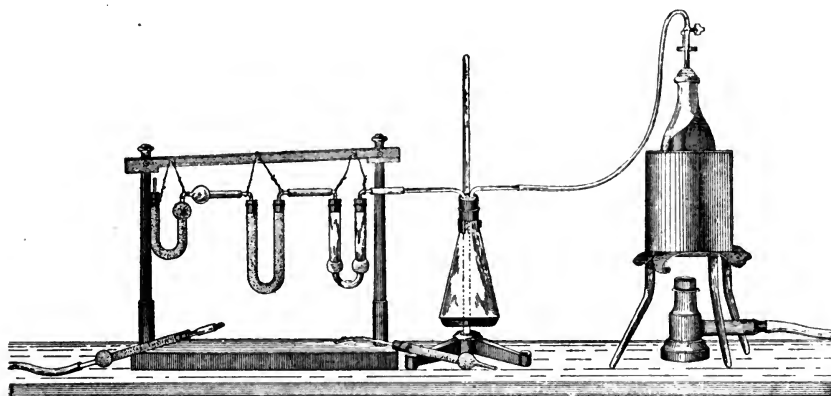


FIG 3.—Apparatus for the determination of carbon dioxide.

When the bottle containing the sample is closed with a patent stopper, the latter may sometimes be replaced by a rubber stopper fitted with stopcock tube. Where the pressure is so great that this is not practicable, such samples may be treated as directed under "Bulk Goods."

## (b) BULK GOODS.

Close a round-bottom flask of about 700-cc capacity with a two-hole rubber stopper fitted with two stopcock tubes bent at right angles—one passing to the bottom of the flask and the other ending just below the stopper.<sup>c</sup> Produce a partial vacuum in the flask by means of an aspirator, and weigh the flask. Dip the end of one of the stopcock tubes below the surface of the beer, or, better, attach it by means of a rubber tube to a champagne tap or small faucet screwed into the cask, and allow about 300 cc of the sample to enter the flask. Weigh the flask and contents, and proceed as directed under "Bottled Goods." Somewhat better results may be

<sup>a</sup> Hassall, Food Adulteration S. & C. Used by Wiley (Am. Chem. Jour., 1886, 8, 200) in the examination of koumiss, and by Crampton in the examination of beer. Crampton found it necessary to regrind the cocks and ream off the thread, leaving a smooth tube. U. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 3, p. 294.

<sup>b</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 3, p. 293.

<sup>c</sup> Windisch (Das chemische Laboratorium des Brauers, p. 247), employs ordinary glass tubes provided with rubber tubing and screw cocks.

obtained by placing a reflux condenser between the flask and absorption apparatus, and heating the flask over a burner to the boiling point. Attach the other stopcock tube to a soda-lime guard tube and pass a current of air through the apparatus. The amount of carbon dioxid is ascertained by the increase in weight of the absorption tube.

#### 16.—DETECTION OF PRESERVATIVES.

Proceed as directed on pages 88 and 107.

### (C) DISTILLED LIQUORS.

#### 1.—DETERMINATION OF SPECIFIC GRAVITY.

Proceed as directed on page 82. Owing to the high alcohol content of distilled liquors, care must be exercised that the temperature at which specific gravity is determined be as nearly 15.6° C. as possible.

#### 2.—DETERMINATION OF ALCOHOL.

Measure 50 cc of the sample (at 15.6° C.) into a distilling flask, dilute with 100 cc of water, and proceed as directed on page 82. The sample of distilled liquor taken for the determination of alcohol is diluted more than in the case of wine because of the errors attending distillates high in alcohol, errors due to evaporation and to making up to volume at temperatures varying slightly from 15.6° C. All measurements must be made at about that temperature.

#### 3.—DETERMINATION OF EXTRACT.

Evaporate 100 cc to sirupy consistency and proceed as directed on page 83.

#### 4.—DETERMINATION OF ASH.

Proceed as directed on page 83.

#### 5.—DETERMINATION OF ACIDITY.

Titrate 100 cc with decinormal sodium hydroxid using phenolphthalein as indicator. The number of cubic centimeters employed is multiplied by 0.0060 for the acidity expressed in grams of acetic acid per 100 cc.

#### 6.—DETERMINATION OF SUGAR.

Proceed as directed on page 85.

#### 7.—DETERMINATION OF FUSEL OIL.<sup>a</sup>

The apparatus recommended for this determination is Bromwell's modification of Roese's fusel-oil apparatus. (See fig. 4.)

The reagents required are fusel-free alcohol that has been prepared by fractional distillation over caustic soda or caustic potash, rejecting the first one-fifth and the last three-fifths of the distillate, and diluted to exactly 30 per cent by volume (sp. gr. 0.96541 at 15.6° C.), chloroform, freed from water and redistilled, and sulphuric acid (sp. gr. 1.2857 at 15° C.).

Distill slowly 200 cc of the sample under examination till about 175 cc have passed over, allow the distilling flask to cool, add 25 cc of water, and distill again till the total distillate measures 200 cc. Dilute the distillate to exactly 30 per cent by volume<sup>b</sup> (sp. gr. 0.96541 at 15.6°).

<sup>a</sup> Windisch, Arb. kais. Gesamt., Vol. V, p. 390.

<sup>b</sup> The following is an accurate method for diluting any given alcohol solution to a weaker solution of definite percentage: Designate the volume percentage of the stronger alcohol by V and that of the



Now prepare a water bath, the contents of which are kept at exactly  $15^{\circ}\text{C}.$ , and place in it the apparatus (covering the end of the tube with a rubber cap to prevent wetting the inside of the tube) and flasks containing the 30 per cent fusel-free alcohol, chloroform, sulphuric acid, and the distillate diluted to 30 per cent by volume. When the solutions have all attained the temperature of  $15^{\circ}\text{C}.$ , fill the apparatus to the 20-cc mark with the chloroform, drawing it through the lower tube by means of suction, add 100 cc of the 30 per cent fusel-free alcohol and 1 cc of the sulphuric acid, invert the apparatus and shake vigorously for two or three minutes, interrupting once or twice to open the stopcock for the purpose of equalizing pressure. Allow the apparatus to stand for one hour in water that is kept at the temperature<sup>a</sup> of  $15^{\circ}\text{C}.$ , turning occasionally to hasten the settling of the chloroform and note the volume of the chloroform. After thoroughly cleansing and drying the apparatus repeat this operation, using the diluted distillate from the sample under examination in place of the fusel-free alcohol. The increase in the chloroform volume with the sample under examination over that with the fusel-free alcohol is due to fusel oil, and this difference (expressed in cubic centimeters) multiplied by the factor 0.663 gives the volume of fusel oil in 100 cc, which is equal to the percentage of fusel oil by volume in the 30 per cent distillate. This must be calculated to the percentage of fusel oil by volume in the original liquor.

*Example.*—A sample of liquor contains 50 per cent of alcohol by volume. The increase in the chloroform volume with the 30 per cent fusel-free alcohol is 1.42 cc. The increase in the chloroform volume with the distillate from the liquor under examination diluted to 30 per cent is 1.62 cc; difference, 0.20 cc. The volume of fusel oil in 100 cc of the 30 per cent distillate, then, is  $0.20 \times 0.663 = 0.1326$ , and by the proportion  $30:50::0.1326:0.221$  we obtain the percentage of fusel oil by volume in the original liquor.

#### 8.—DETERMINATION OF ALDEHYDES.<sup>b</sup>

Dissolve 0.5 grams of fuchsin in about 100 cc of water; add a solution containing the same weight of sulphurous acid ( $\text{H}_2\text{SO}_3$ ); dilute to a liter and filter. With 1 volume of this reagent mix 2 volumes of the 30 per cent distillate obtained in the determination

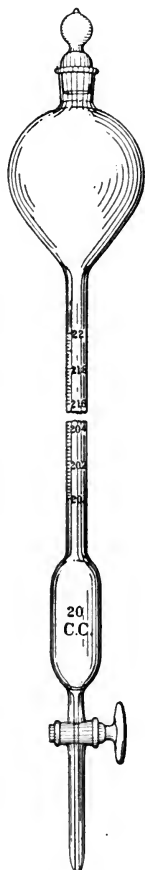


FIG. 4.—Bromwell's fusel-oil apparatus.

weaker alcohol by  $v$ . Mix  $v$  volumes of the stronger alcohol with water to make  $V$  volumes of the product. Allow the mixture to stand till full contraction has taken place and till it has reached the temperature of the original alcohol and water and make up any deficiency in the  $V$  volumes with water.

*Example.*—It is desired to dilute a distillate containing 50 per cent of alcohol by volume until it contains 30 per cent. To 30 volumes of the 50 per cent alcohol add enough water to make 50 volumes, or place 150 cc of the distillate in a 250-cc flask, fill to the mark with water, mix, cool, and fill to the mark again.

Owing to the extreme difficulty of preparing distillates of exactly 30 per cent, slight variations may be corrected by increasing or decreasing the chloroform reading, as suggested by Sell, 0.003 cc for each 0.01 per cent variation in strength of alcohol from 30 per cent. Such variation, however, should not exceed 0.02 per cent.

<sup>a</sup>The temperature must be held as nearly  $15^{\circ}\text{C}.$  as possible. If any variations occur the chloroform must be increased or decreased 0.046 cc for every degree above or below that temperature (Gebek & Stutzer, *Ztschr. ang. Chem.*, 1893, 132).

<sup>b</sup>Medicus Forsch. uber Lebensm., 1895, 2, 299.

of fusel oil. Treat in the same manner and at the same time a solution of 30 per cent (by volume) aldehyde-free alcohol containing 0.05 grams of acetic aldehyde per liter. After two minutes, match the colors of the two mixtures by dilution of the stronger with 30 per cent aldehyde-free alcohol, or by means of a colorimeter, and express the result as acetic aldehyde.

#### 9.—DETERMINATION OF ETHEREAL SALTS.

After the determination of the volatile acids the neutralized distillate is transferred to a flask connected with a reflux condenser, treated with 25 cc of tenth normal sodium hydroxid, and boiled one-half hour. The flask and contents are then cooled, 25 cc of decinormal hydrochloric acid added, and the excess of acid titrated with sodium hydroxid, using phenolphthalein as indicator. The number of cubic centimeters of decinormal alkali used in this titration, multiplied by 0.0088, is equal to the weight in grams of ethereal salts (calculated as ethyl acetate) in the volume of liquor taken for the determination.

#### 10.—DETERMINATION OF FURFUROL.<sup>a</sup>

Treat 5 cc with 5 drops of colorless anilin and 8 drops of acetic acid. After fifteen minutes, compare colorometrically with 5 cc of a solution containing 0.05 grams of furfural per liter which has been subjected to the same treatment.

#### 11.—DETERMINATION OF COLORING MATTER.

Proceed as directed under methods for the detection of coloring matter, (p. 111). For the detection of caramel use the method of Crampton and Simons,<sup>b</sup> which depends on the insolubility of caramel in ether. Evaporate 50 cc of the sample nearly to dryness on the water bath, wash into a 50-cc flask, add 25 cc of absolute alcohol, cool to a definite temperature, and dilute to mark with water. Transfer 25 cc to an apparatus of the general description of Bromwell's fusel-oil apparatus (page 97), but graduated so that the lower bulb holds 25 cc to a definite mark on the stem, which may be of larger tare than in Bromwell's apparatus. Add 50 cc of ether and shake at intervals for half an hour, let settle, and siphon water through the lower tube until the aqueous layer reaches the 25-cc mark. Mix the whole, remove the aqueous layer, and compare by means of a tintometer with the 25 cc of the solution which were not treated with ether. Express the amount of color removed on the percentage basis.

### XV.—BAKING POWDERS AND BAKING-POWDER CHEMICALS.

By A. L. WINTON,

*Chemist of State Experiment Station, New Haven, Conn.*

All the processes hereafter described, except determination of acidity, may be employed in the analysis of baking powders, and all the processes, except determination of carbonic acid, in the analysis of cream of tartar and its substitutes. The sample under examination is entirely removed from the package, carefully mixed, and passed through a sieve without grinding.

#### 1.—DETERMINATION OF TOTAL CARBON DIOXID.<sup>c</sup>

This determination is made by the absorption method, and any apparatus may be employed which gives accurate results when checked with pure calcite. Whatever apparatus is chosen the tubes and materials used for absorbing and drying the carbon dioxide may be varied according to the preference of the analyst. Those

<sup>a</sup> Windisch. *Forsch. über Lebens.*, 1897, 4, 369.

<sup>b</sup> *Jour. Am. Chem. Soc.*, 1890, 22, 810.

<sup>c</sup> See Appendix, p. 156.

mentioned below are selected because the details have been carefully worked out by the originators.

According to the amount of absorbent employed the weight of sodium carbonate or calcium carbonate may vary from 0.25 to 1.00 gram, and about twice as much baking powder may be used. The corrections for temperature and pressure given with the Heidenhain apparatus may ordinarily be disregarded.

(a) KNORR'S APPARATUS.

(1) *Description of apparatus.*

This apparatus (fig. 5) employs only ground-glass joints, and may be quickly made ready for use or taken to pieces and packed away. On the other hand, it is inflexible

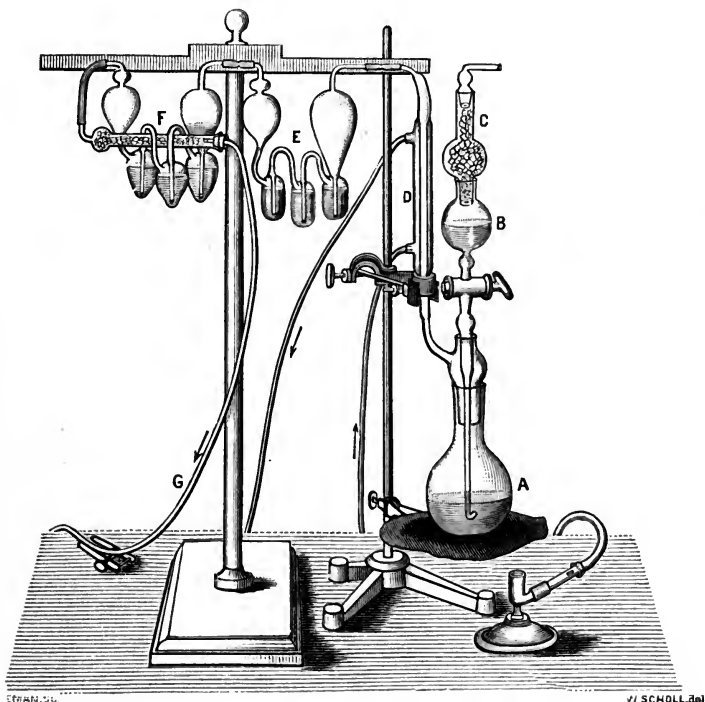


Fig. 5.—Knorr's apparatus for the determination of carbon dioxide: A, Distilling flask fitted to condenser by a ground-glass stopper. B, Reservoir containing acid. C, Soda-lime tube fitted to acid reservoir by a ground-glass stopper. D, Condenser. E, Liebig bulb filled with sulphuric acid. F, Liebig bulb filled with a solution of potassium hydroxid for the absorption of carbon dioxide and followed by a calcium-chlorid tube. An additional guard tube filled with soda lime should follow the tube F, though not shown in the cut.

and must be carefully handled, and has the additional disadvantage that broken parts can not readily be replaced. Therefore it is of more value for occasional determinations than for a long series.<sup>a</sup>

<sup>a</sup>The small calcium chlorid tube shown in the cut attached to the potash bulb F is usually replaced by a second Liebig bulb filled with sulphuric acid. Better results are obtained if the same drying tubes are used before and after the potash bulb. Many analysts prefer to replace the bulb F and attached calcium chlorid tube by two U-tubes filled with sifted soda lime. When the second tube shows a material increase in weight it is placed first, and the first tube refilled and placed in the second position.

(2) *Materials.*

The potassium hydroxid solution usually employed for absorbing carbon dioxid has a specific gravity of about 1.27. Many analysts, however, prefer a solution having a specific gravity of 1.55.

The calcium chlorid and soda lime employed should be finely granulated and freed from dust with a sieve.

(3) *Manipulation.*

The quantity of baking powder to be examined is placed in a distilling flask, which must be perfectly dry.<sup>a</sup> The flask is closed with a stopper carrying the tube connecting with the absorption apparatus and also with the funnel tube. The tubes in which the carbon dioxid is to be absorbed are weighed and attached to the apparatus. In case two Liebig bulbs are employed, one for potassium hydroxid and the other for sulphuric acid, to absorb the moisture given up by the potassium hydroxid solution, it will be necessary to weigh them separately. If two soda-lime tubes are employed it will be found advantageous to weigh them separately and fill the first tube anew when the second tube begins to increase in weight materially. The tube B is nearly filled with hydrochloric acid (sp. gr. 1.1), and the guard tube C placed in position. The aspirator is now started at such a rate that the air passes through the Liebig bulbs at the rate of about two bubbles per second. The stopper of the funnel tube is opened and the acid allowed to run slowly into the flask, care being taken that the evolution of gas shall be so gradual as not to materially increase the current through the Liebig bulb. After the acid has all been introduced, the aspiration is continued, when the contents of the flask are gradually heated to boiling, the bulb in tube B being closed. While the flask is being heated the aspirator tube may be removed, although many analysts prefer when using ground-glass joints to aspirate during the entire operation. The boiling is continued for a few minutes after the water has begun to condense in D, when the flame is removed, the valve in the tube B opened, and the apparatus allowed to cool with continued aspiration. The absorption tubes are then removed and weighed, the increase in weight being due to carbon dioxid.

## (b) HEIDENHAIN'S APPARATUS.

(1) *The apparatus.*

This was originated by G. J. Mulder and recommended and improved by Kolbe, Stolba, and Fresenius,<sup>b</sup> and has been modified by H. Heidenhain,<sup>c</sup> as shown in Fig. 6, which is drawn on a scale of 1 : 12. It consists of—

- A. A cylinder filled with soda-lime to free the air from carbon dioxid. A thick layer of cotton prevents soda-lime dust from being carried over.
- B. Glass cock to regulate the air current, which finds resistance at C.
- C. A capillary contraction.
- D. Funnel tube of peculiar shape. The funnel is cylindrical, three-fourths of an inch wide and 4 inches long, and is reduced to half its width at the bottom, so as to make a neck for a perforated rubber stopper into which—
- E. A glass tube is tightly fitted, allowing the stopper to be taken out and put in by the glass tube.
- F. Evolution flask, ordinarily of 150-cc capacity, for foaming liquids of 300-cc capacity.
- G. Return condenser, simply a glass tube of one-fourth of an inch bore, around which a small lead pipe is wound. The tube following the condenser contains a few pieces of calcium chlorid, to retain the bulk of the moisture. It is refilled when contents are liquefied.

<sup>a</sup> See Appendix, p. 157.

<sup>b</sup> Quant. Anal., vol. 1, p. 449, and vol. 2, p. 308, German edition.

<sup>c</sup> Jour. Am. Chem. Soc., 1896, 18, 1.

- H. U-tube filled with coarse calcium chlorid.
- K. Filled at I with a 3-inch long column of pumice stone impregnated with copper sulphate completely dehydrated at 150° C. The rest is filled with fine calcium chlorid.
- L. Cock to close the apparatus when not in use.
- M. First absorption tube about one-half inch in diameter and 5 inches long, filled mainly with soda-lime, with a little calcium chlorid at the side at which the air current enters.
- N. Second absorption tube of same size as M, filled half with soda-lime and half with calcium chlorid. Place the side containing calcium chlorid toward the end of the apparatus where the air current leaves.
- O. Guard-tube containing calcium chlorid toward N and soda-lime toward P.
- P. Indicator tube trapped with glycerin.
- R. Safety bottle to receive water which may be sucked back from—
- S. The aspirator, which is a Mariotte's bottle of about 4 liters capacity.

### (2) *Materials.*

Use calcium chlorid dehydrated at 200° C, not fused. Grind it coarsely in a coffee mill and sift through No. 18 wire gauze to remove the extremely coarse, and through No. 30 wire gauze to remove the very fine. Prepare a large quantity of such calcium chlorid at the beginning and use this for the tubes K, M, and N. The reason for this is that the current of air must leave the weighed tubes with the same content of moisture as it entered them, which only can be attained if the absorbent in K and N is of the same nature and quality.

The soda-lime\* for the weighed tubes is ground and sifted in the same way. It should not be too dry, as it must not absorb moisture to a higher degree than calcium chlorid. The tubes M and N should hold about 20 grams filling each, making M's capacity for carbon dioxid almost 1 gram and N's capacity for moisture 0.2 gram. M should be refilled when its weight has increased 0.75 gram, and N after an increase of 0.1 gram in weight.

Use best rubber for all connections, applying a trace of castor oil as lubricator. For connections of the weighed tubes use rubber tubing boiled in weak lye, washed and dried. Apply also a little castor oil, which is thoroughly wiped off again before connecting the tubing.

Before using the apparatus fill H and K with carbon dioxid, in order to saturate the alkalinity of the calcium chlorid, and exhaust after several hours.

### (3) *Manipulation.*

Weigh M and N, taking precaution that they are of the same temperature as the air in the balance-room. Shortly before weighing, open the tubes for a moment to allow equalization of air. Note thermometer and barometer. Connect tubes with the apparatus and make sure that all joints are tight by closing A at the bottom, opening all cocks, starting the aspirator and observing P, in which the liquid must soon come to a standstill. Then disconnect the aspirator, close B, remove F, put in the substance<sup>b</sup> (use about 1 gram of sodium carbonate or calcium carbonate, or about 2 grams of baking powder), connect F, and start the cooler. Fill acid and water through D, lifting E slightly and allowing only small quantities of the acid and water to enter at the time. (Use only water made free from carbon dioxid by boiling.) Light the burner, heat to boiling, and reduce the flame to keep the liquid just at the boiling point. If no more air passes P, start the aspiration. When water stops running, open B carefully and adjust the outflow of the aspirator by raising or lowering the syphon to half the safe speed.

\* An excellent method for the preparation of soda-lime is given by Benedict and Turner, Jour. Amer. Chem. Soc., 1899, **21**, 396.

<sup>b</sup> See Appendix, p. 157.

(In order to find the allowable rapidity of the air current proceed as follows: Charge the apparatus exactly as for an analysis, leaving out the carbonate. Start to

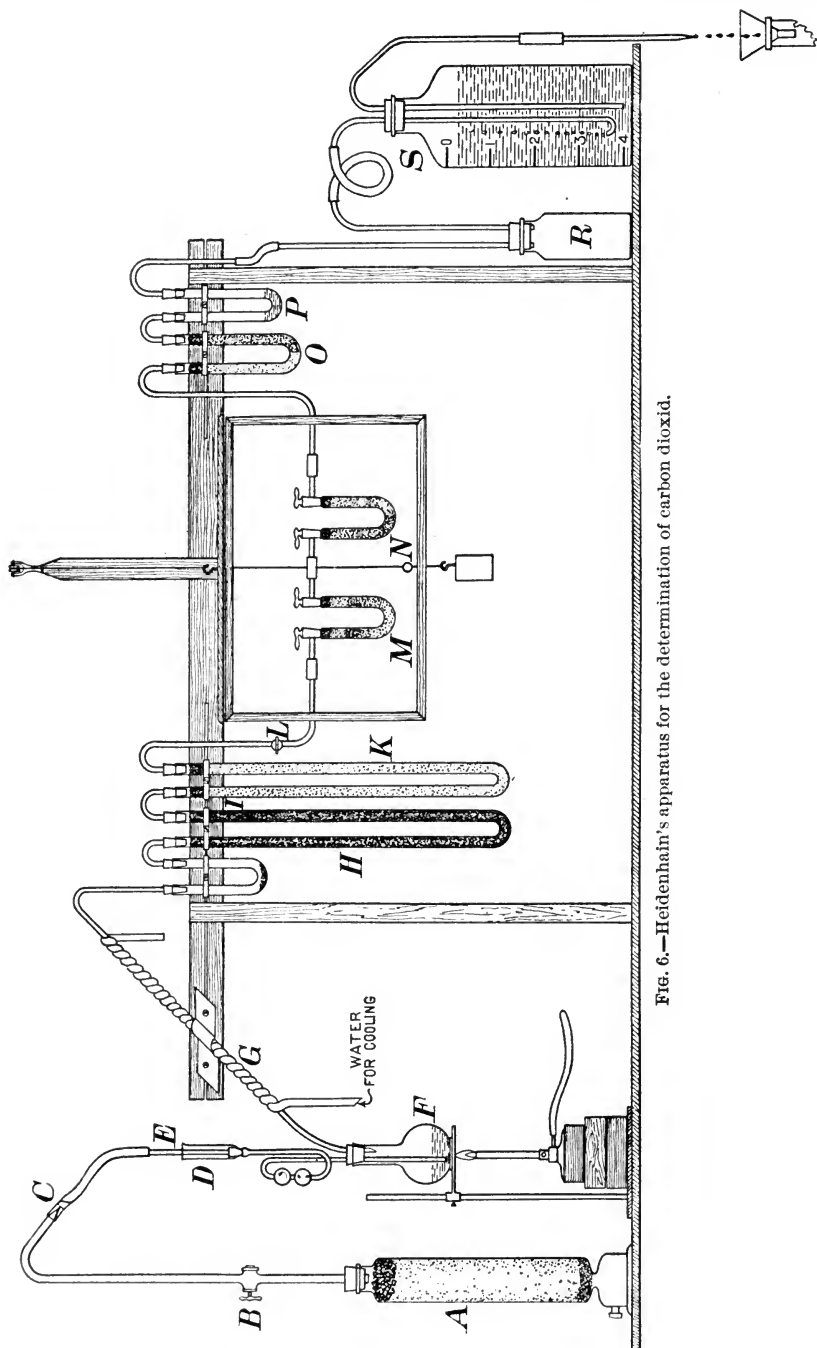


FIG. 6.—Heidenhain's apparatus for the determination of carbon dioxide.

aspirate at the rate of about 50 cc per minute. After two liters have been aspirated weigh the tubes. If they have lost in weight, repeat the experiment with 40 cc per

minute, and so on until the weight of the tubes remains constant. If the work has been done with due precaution, the first tube must have lost just as much as the second has gained. Do not exceed the safe speed thus found.)

After M has become cool increase the current to full safe speed and aspirate altogether 3 liters, continuing boiling to the end of aspiration. After the tubes have assumed the temperature of the balance-room open for a moment, weigh, note again thermometer and barometer, and apply correction according to the following formulæ:

$$-(A^2 - A^1) \times T \text{ and } + (B^2 - B^1) \times B$$

in which

$A^1$  is the temperature at first weighing in  $^{\circ}\text{C}$ ,

$A^2$  is the temperature at second weighing in  $^{\circ}\text{C}$ ,

$B^1$  is the air pressure at first weighing in mm,

$B^2$  is the air pressure at second weighing in mm,

and T and B are constants found as follows:

G = weight of empty tubes.

F = weight of fillings.

The specific gravity of glass = 2.7.

The specific gravity of filling = 2.0.

The specific gravity of brass = 8.5.

Change of weight of 1 cc air with  $1^{\circ} = 0.0000039$  gram.

Change of weight of 1 cc air with 1 mm pressure = 0.0000015 gram.

From above follows:

$$\text{Volume of tubes and fillings} = \frac{G}{2.7} + \frac{F}{2.0}$$

$$\text{Volume of brass weights} = \frac{G+F}{8.5}$$

and

$$\frac{G}{2.7} + \frac{F}{2.0} - \frac{G+F}{8.5} = V,$$

representing the differential volume affected by temperature and pressure and being a constant for the tubes.

Now,

$$T = V \times 0.0000039 \text{ gram, and}$$

$$B = V \times 0.0000015 \text{ gram.}$$

Observe that rise of temperature makes the air lighter, consequently the tubes heavier. Therefore the correction must be negative. On the other hand, increased pressure has the opposite effect, making the correction positive.

Example:

G = 80. F = 40, from which follows

V = 35.5, and

T = 0.00014 gram, and

B = 0.00005 gram.

Now, if  $A^1 = 25^{\circ}$ ,

$A^2 = 27^{\circ}$ ,

$B^1 = 759$  mm,

$B^2 = 756$  mm,

then the correction for temperature will be—

$$-0.00028 \text{ gram,}$$

and for air pressure—

$$-0.00015 \text{ gram,}$$

making a total of—

$$-0.00043 \text{ gram.}$$

2.—DETERMINATION OF RESIDUAL CARBON DIOXID.<sup>a</sup>

Weigh 2 grams of baking powder into a flask suitable for the subsequent determination of carbonic acid, add 20 cc of cold water, and allow to stand 20 minutes. Place the flask in a metal drying cell surrounded by boiling water, and heat with occasional shaking for 20 minutes.

To complete the reaction and drive off the last traces of gas from the semisolid mass, heat quickly to boiling over a lamp, and boil for one minute. Aspirate until the air in the flask is thoroughly changed, and determine the residual carbon dioxide by absorption, as described under *total carbonic acid*.

The process described,<sup>b</sup> based on the methods of McGill<sup>c</sup> and Catlin,<sup>d</sup> imitates as far as practicable the conditions encountered in baking, but in such a manner that concordant results may be readily obtained on the same sample, and comparable results on different samples.

## 3.—DETERMINATION OF AVAILABLE CARBON DIOXID.

Subtract the residual carbon dioxide from the total.

## 4.—DETERMINATION OF ACIDITY.

(For cream of tartar and its substitutes.)

Dissolve one gram of the material in hot water and titrate with standard fifth-normal potassium hydroxide solution, using phenolphthalein as indicator.

5.—DETECTION OF TARTARIC ACID, FREE OR COMBINED.<sup>e</sup>

*Applicable in presence of phosphates.*

Shake repeatedly about 5 grams of the sample with about 250 cc of cold water in a flask and allow the insoluble portion to subside. Decant the solution through a filter and evaporate the filtrate to dryness. To the dry powdered residue add a few drops of a 1 per cent solution of resorcin and about 3 cc of strong sulphuric acid. Heat slowly. A rose-red color indicates tartaric acid, the color being discharged on dilution with water.

## 6.—DETECTION OF FREE TARTARIC ACID.

Extract 5 grams of the powder with absolute alcohol and evaporate the alcohol from the extract. Dissolve the residue in dilute ammonia, transfer to a test tube, add a good-sized crystal of silver nitrate, and heat gently. Tartaric acid is indicated by the formation of a silver mirror. If desired, the absolute alcohol extract may be tested by the Wolff method, as described under 5.

7.—DETERMINATION OF TOTAL TARTARIC ACID.<sup>f</sup>

The following is the Goldenberg-Geromont-Heidenhain method, applicable only in the absence of aluminum salts, calcium salts, and phosphates:

Into a shallow porcelain dish, 6 inches in diameter, weigh out 2 grams of the material and sufficient potassium carbonate to combine with all tartaric acid not in the form of potassium bitartrate. Mix thoroughly with 15 cc of cold water and add 5 cc of 99 per cent acetic acid. Stir for half a minute with a glass rod bent near the end. Add 100 cc of 95 per cent alcohol, stir violently for 5 minutes and allow to settle at least 30 minutes. Filter on a Gooch crucible with a thin layer of paper pulp, and wash with 95 per cent alcohol until 2 cc of the filtrate do not change the

<sup>a</sup> See Appendix, p. 157.

<sup>b</sup> Conn. Agr. Exp. Sta. Rep., 1900, p. 169.

<sup>c</sup> Lab. Inland Rev. Dept., Ottawa, Canada, Bul. 68, p. 31.

<sup>d</sup> Baking powders. A Treatise on the Character, Methods for Determination of the Values, etc., p. 20.

<sup>e</sup> Wolff, Rev. chim. anal. appl., 1899, 4, 263.

<sup>f</sup> See Appendix, p. 158.



color of litmus tincture diluted with water. Place the precipitate in a small casserole, dissolve in 50 cc of hot water and add standard fifth-normal potassium hydroxid solution, leaving it still strongly acid. Boil for one minute. Finish the titration, using phenolphthalein as indicator and correct the reading by adding 0.2 cc. One cubic centimeter of fifth-normal potassium hydroxid solution is equivalent to 0.026406-gram tartaric anhydrid ( $C_4H_4O_5$ ), 0.03001 gram tartaric acid ( $H_2C_4H_4O_6$ ), and 0.03763 gram potassium bitartrate ( $KHC_4H_4O_6$ ).

The standard of the potassium hydroxid solution should be fixed by pure dry potassium bitartrate.

The accuracy of this method is indicated by the agreement of the percentages of potassium bitartrate in cream of tartar powders containing no free tartaric acid, obtained by calculation from the tartaric acid, with those obtained by calculation from the potassium oxide.<sup>a</sup>

#### 8.—DETERMINATION OF STARCH.

##### (a) DIRECT INVERSION METHOD.

(For all baking powders and baking chemicals free from lime.)

Weigh 5 grams of the powder into a graduated 500 cc flask. Convert into dextrose by the Sachsse's method and determine the reducing power of the solution by the Allihn method, as described under Spices (p. 57).

##### (b) INDIRECT METHOD.<sup>b</sup>

(For phosphate, alum phosphate, and all other baking powders containing lime.)

Mix 5 grams of the powder in a graduated 500-cc flask, with 200 cc of 3 per cent hydrochloric acid, and allow the mixture to stand for one hour, with frequent shaking. Filter on a Schleicher and Schuell No. 575 11 cm hardened filter, taking care that a clear filtrate is obtained. Rinse the flask once, without attempting to remove all the starch, and wash the paper twice with cold water. Carefully wash the starch from the paper back into the flask, with 200 cc of water, using a small wash bottle. Add 20 cc of 25 per cent hydrochloric acid and proceed according to Sachsse's method. Determine reducing power by Allihn's method.

The treatment with 3 per cent hydrochloric acid, without dissolving the starch, effectually removes the lime, which otherwise would precipitate as tartrate in the alkaline copper solution

##### (c) M'GILL METHOD.

The following modification of McGill's method is valuable for check purposes:

Digest one gram of the powder with 150 cc of 3 per cent hydrochloric acid for 24 hours at the room temperature, with occasional shaking. Filter on a Gooch crucible, wash thoroughly with cold water and finally once with alcohol and once with ether. Dry at 110° C. (4 hours is usually sufficient), cool and weigh. Burn off the starch and weigh again. To obtain the weight of starch subtract the weight after burning from the weight after drying at 110° C.

The results by this method on cream of tartar powders and tartaric acid powders agree closely with those obtained by copper reduction. On phosphate, alum, and alum-phosphate powders the results are usually satisfactory, but in some instances they may be over 2 per cent too high.

#### 9.—DETERMINATION OF POTASSIUM BITARTRATE.

If, as is usually the case, no other potassium salt but the bitartrate is present, multiply the percentage of total potash determined as directed under 12, d, by 3.9936.

<sup>a</sup> Conn. Agr. Exp. Sta. Rep., 1900, p. 180.

<sup>b</sup> After Winton, Conn. Agr. Exp. Sta. Rep., 1900, p. 174.

## 10.—DETERMINATION OF FREE TARTARIC ACID.

Calculate the percentage of tartaric anhydrid combined with the potash as bitartrate (if any) and subtract this from the percentage of total tartaric anhydrid. The difference is the tartaric anhydrid originally added as the free acid, although if the sample has been kept for a long time or has been improperly stored, a portion or all of this acid may exist at the time of analysis as the sodium salt resulting from the reaction in the can with the sodium bicarbonate. Multiply by 1.1365 to obtain the percentage of tartaric acid.

11.—DETECTION OF ALUM IN PRESENCE OF PHOSPHATES.<sup>a</sup>

## (a) IN BAKING POWDER.

Burn to an ash about 2 grams of the sample in a platinum dish. Extract with boiling water and filter. Add to the filtrate a few drops of ammonium chloride solution. A flocculent precipitate indicates alum.

## (b) IN CREAM OF TARTAR.

Mix about 1 gram of the sample with an equal quantity of sodium carbonate, burn to an ash, and proceed as in (a).

12.—EXAMINATION OF ASH.<sup>b</sup>

## (a) DETERMINATION OF INSOLUBLE ASH AND PREPARATION OF SOLUTIONS:

Char 5 grams of the material in a platinum dish at a heat below redness. Boil the carbonaceous mass with dilute hydrochloric acid, filter into a graduated 500-cc flask, and wash with hot water. Return the residue, together with the paper, to the platinum dish and burn to a white ash. Boil again with hydrochloric acid, filter, wash, unite the two filtrates, and dilute to 500 cc.

Incinerate the residue after the last filtration for the determination of ash insoluble in acid.

(b) IRON AND ALUMINA.<sup>c</sup>

Draw an aliquot portion of 100 cc and separate silica, if necessary. Mix the solution with sodium-phosphate solution in excess of what is required to form normal aluminum phosphate. Add ammonia until a precipitate remains on stirring, then hydrochloric acid drop by drop until the precipitate dissolves. Heat the solution to about 50° C., mix with a considerable excess of 50 per cent ammonium-acetate solution and 4 cc of 80 per cent acetic acid.

As soon as the precipitate of aluminum phosphate, mixed with a little iron phosphate, has settled, collect on a filter, wash with hot water, ignite, and weigh.

Fuse the mixed phosphates with ten parts of sodium carbonate, dissolve in dilute sulphuric acid, reduce with hydrogen sulphid and determine the iron by the volumetric permanganate method. In the same solution determine the phosphoric acid. To obtain the weight of  $\text{Al}_2\text{O}_3$ , subtract the sum of the weights of  $\text{Fe}_2\text{O}_3$  and  $\text{P}_2\text{O}_5$  from the weight of the mixed phosphates.

## (c) LIME.

Heat the filtrate from the mixed phosphates, which is acid with acetic acid, to 50° C. and precipitate with ammonium oxalate. Filter, wash, ignite over a Bunsen burner, and finally convert into oxid by heating over a blast lamp.

<sup>a</sup>Thirty-first An. Rep. Mass. State Board of Health, 1899, p. 638.

<sup>b</sup>Conn. Agr. Exp. St., Rep. 1900, p. 178.

<sup>c</sup>See Appendix, p. 160.

(d) POTASH AND SODA.<sup>a</sup>

Evaporate an aliquot portion of the solution, prepared as described, nearly to dryness to remove the excess of hydrochloric acid, dilute, and heat to boiling. While still boiling, add barium chloride solution as long as a precipitate forms and enough barium hydrate to make the liquid strongly alkaline. As soon as the precipitate has settled, filter and wash with hot water, heat the filtrate to boiling, add sufficient ammonium carbonate solution (1 part of ammonium carbonate in 5 parts of 2 per cent ammonia water) to precipitate all the barium, filter, and wash with hot water. Evaporate the filtrate to dryness, ignite below redness to remove ammonia salts. Add to the residue a little water and a few drops of ammonium carbonate solution. Filter into a tared platinum dish, evaporate, ignite below redness, and weigh the mixed potassium and sodium chlorides.

Determine the potash as potassium platinichlorid, using the factors 0.1939 for  $K_2O$  and 0.3069 for  $KCl$ .

## 13.—DETERMINATION OF PHOSPHORIC ACID.

Mix 5 grams of the material with a little magnesium-nitrate solution, dry, ignite, and dissolve in hydrochloric acid. In an aliquot of the solution determine phosphoric acid as magnesium pyrophosphate by the molybdic method.<sup>b</sup>

## 14.—DETERMINATION OF SULPHURIC ACID.

Boil 5 grams of the powder gently for one and one-half hours with a mixture of 300 cc of water and 15 cc of concentrated hydrochloric acid. Dilute to 500 cc, draw off an aliquot portion of 100 cc, dilute considerably, precipitate with barium chlorid, filter through a Gooch crucible, ignite, and weigh. Direct solution of the material without burning of the organic matter was proposed by Crampton.<sup>c</sup> The dextrose, formed by the action of the acid on the starch of baking powders, does not interfere with the accuracy of the process.<sup>d</sup>

## 15.—DETERMINATION OF AMMONIA.

Ammonia alum is often an ingredient of cream-of-tartar substitutes and baking powders, and ammonium carbonate is occasionally present in baking powders. Determine ammonia by distillation with caustic soda into standard acid and titration.

**XVI. FOOD PRESERVATIVES.**

By W. M. ALLEN,

*State Department of Agriculture, Raleigh, N. C.*

## 1.—DETECTION OF FORMALDEHYDE.

## (a) PREPARATION OF SAMPLE.

If the material be solid or semisolid, macerate from 200 to 300 grams in a mortar with about 100 cc of water until a sufficient degree of fluidity is obtained. Take the sample so prepared and make distinctly acid with phosphoric acid. Transfer to a short-necked distilling flask of copper or glass of from 500 to 800-cc capacity. If a copper flask is used, the heat can be applied directly; if the flask be glass, it is best to heat in a linseed-oil bath. Connect flask with glass condenser and distill off from 40 to 50 cc. In the case of liquids, acidify from 200 to 300 cc with a strong excess of phosphoric acid, and distill as directed under 3.

<sup>a</sup>See Appendix, p. 160.

<sup>b</sup>U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 12.

<sup>c</sup>U. S. Dept. of Agr., Div. of Chem., Bul. 13, part 5, p. 596.

<sup>d</sup>Conn. Agr. Expt. Sta., Rep. 1900, p. 119.

## (b) FIRST METHOD OF DETECTION.

To about 5 cc of the distillate described above add 2 or 3 drops of a 1 per cent aqueous solution of phenol; mix and carefully pour it on about the same amount of concentrated sulphuric acid in a test tube, holding the tube so that the solutions will not mix. The presence of one part of formaldehyde in 100,000 parts is indicated by the formation of a crimson color at the plane of union of the solutions. If the formaldehyde be present in greater quantity, a white turbidity, or a light-colored precipitation, will be formed above the coloring.

If organic matter is distilled over, the charring of it by the sulphuric acid may be mistaken for a trace of formaldehyde; but, on allowing the test to stand for twelve hours, the coloration, if due to formaldehyde, will become a whitish turbidity instead of the dark color which appears if due to the charring of organic matter. Some other aldehydes will give the same result, and it is, therefore, not conclusive.

(c) SECOND METHOD OF DETECTION.<sup>a</sup>

Add about 5 cc of the distillate obtained under (a) to an equal volume of pure milk in a porcelain casserole, and about 10 cc of concentrated hydrochloric acid containing 1 cc of 10 per cent ferric chlorid solution to each 500 cc of acid. Heat to 80° or 90° directly over the gas flame, holding the casserole by the handle and giving it a rotary motion to break up the curd. A violet coloration indicates formaldehyde.

## (d) THIRD METHOD OF DETECTION.

Dissolve 1 gram of phenylhydrazin hydrochlorid and 1.5 grams of sodium acetate in 10 cc of water. To 1 cc of distillate obtained as directed in (d) add 2 drops of reagent and 2 drops of sulphuric acid. If formaldehyde is present, a green color will be produced.

## 2.—DETECTION OF SULPHUROUS ACID.

Prepare samples as directed under 1 (a), and boil 20 cc of the distillate after the addition of a few drops of bromin or iodine solution. If it is decolorized quickly, test for sulphuric acid with barium chlorid solution.

If sulphurous acid or sulphite is present, determine it quantitatively as directed under wine (page 90).

## 3.—DETECTION OF SALICYLIC ACID.

If the material be a solid or semisolid, macerate 200 to 300 grams in a mortar with about 400 cc of water made slightly alkaline with sodium or potassium hydroxid, and strain through a cotton bag. Acidify the filtrate with dilute (1:3) sulphuric acid, and extract by shaking with about 30 cc of chloroform or ether.<sup>b</sup> Separate from the water with a separatory funnel. If a clear solution is obtained, place the chloroform or ether in a small porcelain dish and evaporate at a low temperature. If an emulsion is formed and a clear solution will not separate out on standing, whirling in a Babcock milk tester or some other centrifugal machine will usually give the desired clear solution. Take up the residue in the porcelain dish with 3 or 4 cc of hot water and divide into two portions, one for salicylic acid and the other for saccharin.

In the case of materials containing large amounts of extractive matter, and those from which the water solution can not be separated from the solid matter by straining, it may be found necessary to separate them by distillation, though straining is always preferable. In such cases acidify the macerated material with phosphoric acid, and transfer to a distilling flask, with a very short neck and wide mouth. An Erlenmeyer flask with inside diameter of mouth  $1\frac{1}{4}$  inches is a good shape. The

<sup>a</sup> Leach, Twenty-ninth An. Rep. Mass. Board of Health, 1897, p. 558.

<sup>b</sup> See appendix, page 160.

tube connecting the flask with condenser should be very short, with an inside diameter of not less than  $\frac{3}{8}$  inch, and should turn into the condenser immediately above the stopper in flask. Conduct steam through a small tube passing through the stopper and dipping deeply into the material in the flask. Submerge the distilling flask almost to the stopper in a linseed oil bath and distill with temperature of the oil at from 120° to 130° C.

Care must be taken not to let the contents of the flask get too low, as the heat will decompose the organic matter. The temperature in the flask will go but little above 100° C. unless the solution in the flask is allowed to get too low.

Distill off 500 cc to 600 cc, acidify with dilute sulphuric acid, extract with about 30 cc of chloroform or ether, and proceed as directed above.

(a) SACCHARIN.

If the solution for saccharin and salicylic acid has an intensely sweet taste, it is an indication of saccharin. If it is sweet, dilute a portion of it about ten times, and taste again. (See also page 51.)

(b) SALICYLIC ACID.

(1) *First method.*<sup>a</sup>

Place a few drops of solution for salicylic acid in a porcelain dish, add 2 or 3 drops of ferric chlorid solution in such a way that the solutions will come together slowly, which will give a purple or violet color if salicylic acid is present.

(2) *Second method.*<sup>a</sup>

Place about 0.5 cc of the solution in a porcelain dish and evaporate to dryness at a low temperature. Warm the residue carefully with one drop of concentrated nitric acid, and add 2 or 3 drops of ammonia until alkaline. The presence of salicylic acid is indicated by the formation of a yellow color of ammonium picrate, and may be confirmed by dyeing a thread of fat-free wool in it.

4.—DETECTION OF BENZOIC ACID.<sup>b</sup>

Separate benzoic acid by extraction or distillation as directed under salicylic acid, and test by one of the following methods:

(1) *First method.*<sup>c</sup>

Divide solution for benzoic acid into three portions and examine one portion by each of the following methods: Make alkaline with ammonium hydroxid, expel the excess of ammonia by evaporation, take up the residue with water, and add a few drops of a neutral 0.5 per cent solution of ferric chlorid. The presence of benzoic acid will be indicated by the formation of a brownish-colored precipitate of ferric benzoate.

(2) *Second method.*<sup>c</sup>

Evaporate to dryness and treat the residue with 2 or 3 cc of strong sulphuric acid, Heat till white fumes appear; organic matter is charred and benzoic acid is converted into sulpho-benzoic acid. A few crystals of potassium nitrate are then added. This causes the formation of metadinitrobenzoic acid. When cool, the acid is diluted with water and ammonia added in excess, followed by a drop or two of ammonium

<sup>a</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 51, p. 132.

<sup>b</sup> See also Appendix, page 160.

<sup>c</sup> Bul. Soc. Chim. 1890 [3], 3, 414.

sulphid. The nitrocompound becomes converted into ammonium metadiamidobenzoic acid, which possesses a red color. This reaction takes place immediately, and is seen at the surface of the liquid without stirring.

(3) *Third method.*

Evaporate to dryness at low temperature. If benzoic acid is present in great quantity it will crystallize out in shining leaflets, with characteristic odor. These will melt at 120° C.

5.—DETECTION AND DETERMINATION OF BORIC ACID AND BORATES.

(a) FIRST QUALITATIVE METHOD.<sup>a</sup>

Render decidedly alkaline with lime water about 25 grams of the sample and evaporate to dryness on a water bath. Ignite the residue to destroy organic matter. Add about 15 cc of water and hydrochloric acid, drop by drop, to acid reaction. Then add about 1 cc of concentrated hydrochloric acid. Moisten a piece of delicate turmeric paper with the solution; if borax or boric acid is present, the paper on drying will acquire a peculiar red color, which is changed by ammonia to a dark blue-green, but is restored by acid. This color is almost unmistakable, but it is best for one not familiar with it to conduct a blank.

A preliminary test may be made by immersing a strip of turmeric paper in about 100 cc of liquid foods, to which about 7 cc of concentrated hydrochloric acid has been added. Solid and pasty foods may be heated with enough water to make them thoroughly fluid, hydrochloric acid added in about the proportion of 1 to 15, and tested in the same manner.

(b) SECOND QUALITATIVE METHOD.<sup>b</sup>

Add an equal volume of fresh saturated turmeric tincture and a drop of hydrochloric acid, and heat a few seconds. If the sample is a liquid, evaporate with the turmeric and heat with a drop of dilute hydrochloric acid for a few seconds; then if borax or boric acid is present a pink or dark red color will appear, depending upon the amount of boric acid present. Cool and add a drop of ammonium hydroxid, when a dark blue-green will appear.

(c) QUANTITATIVE METHOD.<sup>c</sup>

Render 100 grams of the sample decidedly alkaline with sodium hydroxid, and evaporate to dryness in a platinum dish. Ignite the residue cautiously, heat with about 20 cc of water, and add hydrochloric acid, drop by drop, until all is dissolved. Transfer to 100-cc flask, the bulk not being allowed to go over 50 cc or 60 cc. Add 0.5 gram of calcium chlorid and a few drops of phenolphthalein, then a 10 per cent solution of caustic soda until a permanent slight pink color is produced, and finally 25 cc of lime water. Make the volume up to 100 cc. Mix well and filter through a dry filter. To 50 cc of the filtrate add normal sulphuric acid till the pink color disappears, then methyl orange, and continue the addition of the acid until the yellow is just changed to pink. Then add fifth-normal caustic soda till the liquid assumes the yellow tinge, excess of soda being avoided. Boil to expel carbon dioxid. Cool the solution, add a little phenolphthalein, and an equal volume of glycerine. Titrate with standardized sodium hydroxid until a permanent pink color is produced.

One cubic centimeter of fifth-normal soda solution is equal to 0.0124 gram crystallized boric acid.

<sup>a</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 51, p. 134.

<sup>b</sup> U. S. Dept. of Agr., Div. of Chem., Bul. 51, p. 113.

<sup>c</sup> Thomson's method—Sutton's Volumetric Analysis, page 100.

## XVII.—COLORING MATTER.

By L. M. TOLMAN,

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## 1.—GENERAL DISCUSSION.

The food chemist has two problems in connection with coloring matter—the analysis of dyes used for food colors and the detection and identification of the color used in a food. The first will require an estimation of the heavy metals present and a determination of the general group to which the color belongs. The second will require the detection of the presence of the color, the determination of the group to which the color belongs, and the presence or absence of poisonous metals.

The complete examination of dyes is too large a subject to take up in these methods, and one will have to refer to such works as Schultz and Julius, on Organic Coloring, Allen's Commercial Organic Analysis, and others that go into the subject in an exhaustive manner. The determination of the general nature of the dye can be made by the use of Rota's scheme, which is the simplest of the many different methods proposed and is quite satisfactory, although it requires a great deal of care and experience.

The detection of the color in a food product and its identification are more difficult. It must be separated in a somewhat pure condition and then tested. Almost all the methods for separating added color from the food will take up some of the natural color of the food as well.

As will be seen in tables for the extraction of fruit colors (p. 113), amyl alcohol extracts the coloring matter from many fruits, and these extracts may easily be mistaken for added colors.

Some of the highly colored fruit juices will dye wool, and the color will be permanent; but these will not be mistaken for coal-tar dyes if the double-dyeing method is followed.

In the methods of manufacture of coal-tar dyes many become contaminated with poisonous metals, such as arsenic, copper, zinc, tin, and lead. There is always the possibility of the presence of arsenic, as sulphuric acid is used at one stage or another in the preparation of nearly every dye.

Some colors have metallic atoms in their molecule, such as malachite green, which is a double chlorid of zinc in combination with the organic group.

Many vegetable colors are sold as lakes of tin or alum. Other colors are known to have a toxic action, such as picric acid and naphthol yellow.

Mixtures of two or more dyes are often added to foods. This can sometimes be shown by a system of fractional dyeing, where the dyes are taken up at different rates by the fabric. In examining mixtures of red, orange, and blue dyes, which are widely sold for coloring wine, the writer found that the woollen cloth took up the red much faster than the orange, and the blue slowest; so that the first piece of cloth dyed was red; the second, a lighter shade; the third, greenish, and the fourth, bluish.

## 2.—DETERMINATION OF HEAVY METALS.

Directions for this determination are given under Vegetables (p. 52).

## 3.—DETERMINATION OF COAL-TAR COLORING MATTERS BY DYEING WOOL.

(a) METHOD OF SOSTEGNI AND CARPENTIERI.<sup>a</sup>

From 10 to 20 grams of the sample are dissolved in 100 cc of water, filtered if necessary, acidified with from 2 to 4 cc of 10 per cent solution of hydrochloric acid, and a piece of woollen cloth, which has been washed in a very dilute solution of boiling

<sup>a</sup> Ztsch. anal. Chem., 1896, 35, 397; U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 68.

potassium hydroxid and then washed in water, is immersed in it and boiled for five to ten minutes. The cloth is removed, thoroughly washed in water, and boiled with very dilute hydrochloric acid solution. Then after washing out the acid the color is dissolved in a solution of ammonium hydroxid (1 to 50). With some of the dyes solution takes place quite readily, while with others it is necessary to boil some time. The wool is taken out, a slight excess of hydrochloric acid is added to the solution, another piece of wool is immersed and again boiled. With vegetable coloring matter this second dyeing gives practically no color, and there is no danger of mistaking a fruit color for one of coal-tar origin. It is absolutely necessary that the second dyeing should be made, as some of the coal-tar dyes<sup>a</sup> will dye a dirty orange in the first acid bath which might be easily passed for vegetable color, but on solution in alkaline bath the second acid bath dyes a bright pink.

(b) ARATA'S METHOD.<sup>b</sup>

This method gives results comparable with those of the first dyeing of the preceding method. It was recommended for detecting coal-tar colors in wine, and has been used by Winton<sup>c</sup> in fruit products.

From 20 to 30 grams of the sample dissolved in 100 cc of water are boiled for ten minutes with 10 cc of a 10 per cent solution of potassium bisulphate and a piece of white wool or woollen cloth which has been previously heated to boiling in a very dilute solution of sodium hydroxid and thoroughly washed in water. After removal from the solution the wool is washed in boiling water, and dried between filter papers. If the coloring matters are entirely from the fruit the wool will be either uncolored or will take on a faint pink or brown, which is changed to green or yellow by ammonia and not restored by washing.

In addition to this, it is advisable in all cases to dissolve out the coloring matter with ammonia as in the first method and dye again, since Arata's method gives practically the same results as the first dyeing in hydrochloric acid bath and needs to be substantiated by the second dyeing.

Another advantage in the second dyeing is that if a large piece of woollen cloth is used in the first dyeing, and a small piece in the second dyeing, small amounts of coloring matter can be brought out much more decidedly in the second dyeing where practically all of the vegetable coloring matter has been excluded. The coloring matter can be identified to a certain extent by the schemes of Witt,<sup>d</sup> Allen, Weingartner,<sup>e</sup> Dommergue,<sup>f</sup> Girard<sup>g</sup> and Dupre, and Rota.<sup>h</sup> The tests can be made<sup>i</sup> directly on the dyed fabric or the dye can be dissolved out.<sup>j</sup> To remove the color wash the wool with dilute tartaric acid and then with water and dry between filter paper. Saturate the wool with strong sulphuric acid and press out the color with a glass rod after from five to ten minutes and dilute to 10 cc with water.

Remove the wool, make solution alkaline with ammonia, and when cold extract with from 5 to 10 cc of amyl alcohol. Separate the amyl alcohol, evaporate it to dryness, and test the residue with strong sulphuric acid.

Ponceau R, 2R, 3R, S and 3S gives yellow red to carmine red.

Ponceau S and tropaeolin O give yellow to orange yellow.

<sup>a</sup> U. S. Dept. of Agr., Bureau of Chem. Bul. 66.

<sup>b</sup> Ztschr. anal. Chem., 1889, **28**, 639.

<sup>c</sup> Conn. Exp. Sta. Report, 1899, Pt. II, p. 131.

<sup>d</sup> Ztschr. anal. Chem., 1887, **26**, 100.

<sup>e</sup> Com. Org. Anal., Vol. III, pt. 1, pp. 399-420.

<sup>f</sup> Ztsch. anal. Chem., 1888, **27**, 232-249.

<sup>g</sup> Ztsch. anal. Chem., 1890, **29**, 369-377.

<sup>h</sup> Analyse des Matières Alimentaires, etc., 583-593.

<sup>i</sup> Analyst, 1899, **24**, 41.

<sup>j</sup> Ztsch. anal. Chem., 1889, **28**, 639; Borgmann, Anal. des Weines, p. 91; Winton, Conn. Expt. Sta. Rept., 1899, Pt. II, p. 131.



Biebrich scarlet gives a green; Bordeaux red and crocein scarlet give blue; tropaeolin 000 and solid red give violet.

If the wool is well dyed most of these colors may be obtained on the fabric.

This gives only the reactions of a few of the more common colors. In order to carry the work farther the more complete works referred to will have to be used.

#### 4.—DETECTION OF COAL-TAR COLORS BY EXTRACTION WITH SOLVENTS.

In the Paris Municipal Laboratory<sup>a</sup> the following scheme of extraction of coal-tar colors is used:

The acid colors, sulphu-fuchsin, azo derivatives, and phthaleins are not precipitated by tannin and are insoluble or only slightly soluble in acetic ether or amyl alcohol.

The basic colors (fuchsin, safranin, etc.) are precipitated by tannin and readily soluble in acetic ether or amyl alcohol.

I. To 50 cc of wine add ammonium hydroxid in slight excess; then add 15 cc of amyl alcohol, shake, and allow to stand.

1. If the alcohol be colored red or violet, decant, wash, filter, evaporate to dryness in presence of a piece of wool, and test the dyed wool with sulphuric acid.

2. If the alcohol be not colored, separate, and add acetic acid. If the alcohol becomes colored the presence of basic aniline color is indicated.

3. If the amyl alcohol is uncolored, both before and after the addition of acetic acid, no basic coal-tar color is present.

II. Add an excess of calcined magnesia and then a 20 per cent solution of mercuric acetate and bring to a boil. A coloration before or after addition of acetic acid indicates the presence of coal-tar dyes, particularly acid dyes.

III. Extract the solution with acetic ether made alkaline by barium hydroxid. This dissolves basic colors.

In any case the colors must be fixed on wool, as many of the fruit colors are extracted and will give reactions with sulphuric acid, which may be mistaken for coal-tar colors.

The extraction of fruit colors is shown in the following tables, the first of which was prepared by Truchon and Martin-Claude,<sup>b</sup> and the second by the writer. The fresh fruit juice was very slightly acidified by hydrochloric acid before extraction. In no case in the dyeing test was there any danger of mistaking the vegetable color for one of coal-tar origin where the double-dyeing method was used.

#### *Extraction of fruit colors with amyl-alcohol.*

Fruit.	Coloration of acid solution. <sup>c</sup>		Coloration of ammoniacal solution.		Addition of a drop of H <sub>2</sub> SO <sub>4</sub> to dyed fabric.
	Juice.	Amyl-alcohol extract.	Juice.	Amyl-alcohol extract.	
Early cherries .....	Red .....	Yellow .....	Green .....	Uncolored .....	Yellow. Yellow. Rose. Rose (dyes silk a rose red).
Ripe cherries .....	Red .....	Uncolored ..	Green .....	Uncolored .....	
Early strawberries .....	Red .....	Rose .....	Green .....	Uncolored .....	
Ripe strawberries .....	Red .....	Red .....	Green .....	Uncolored .....	
Raspberries .....	Red .....	Red .....	Green .....	Uncolored .....	Dyes silk rose. Uncolored.
Ripe currants .....	Red .....	Uncolored ..	Green .....	Uncolored .....	
White currants .....	White .....	Uncolored ..	Brown .....	Uncolored .....	
Black currants .....	Dark red ..	Red .....	Deep green ..	Uncolored .....	
Peaches .....	Yellow .....	Uncolored ..	Brown .....	Yellow-red ..	
Pears .....	Yellow .....	Uncolored ..	Brown .....	Yellow-red ..	
Quinces .....	Yellow .....	Uncolored ..	Brown .....	Yellow-red ..	
Apples .....	Yellow .....	Uncolored ..	Brown .....	Yellow-red ..	
Apricots .....	Yellow .....	Uncolored ..	Brown .....	Yellow-red ..	
Green gage plums .....	Yellow .....	Uncolored ..	Brown .....	Yellow-red ..	

<sup>a</sup> Girard and Dupré *Analyse des Matières*, etc., p. 167

<sup>b</sup> Journ. pharm. chim., 1901, 13, 174.

<sup>c</sup> Acidity of the juice.

*Extraction of fruit colors with amyl-alcohol and with ether.*

Fruit.	Color with NH <sub>4</sub> OH.	Ether ex- tract from acid solution.	Amyl-alcohol extract from acid solution.	Dyeing tests on the juice.
Strawberry .....	Purple.....	None .....	Deep red .....	Color washed out.
Red raspberry .....	Purple.....	None .....	Deep red .....	All color does not wash out, but does not dye in the second acid bath.
Blackberry .....	Blue-purple ..	None .....	Very deep red.	Dyes purplish red in acid solution, but does not dye in the second acid bath.
Cherry.....	Purple.....	None .....	Red .....	Do.
Blackberry .....	Blue-purple ..	None .....	Red .....	Do.
Wild dewberry .....	Blue-purple ..	None .....	Red .....	Do.
Currant.....	Blue-purple ..	None .....	Red .....	Do.

It will be seen from these two tables that amyl-alcohol, as a rule, extracts fruit coloring matter from acid solution, while ether does not. Neither amyl-alcohol nor ether extracted any color from alkaline solution of the fruit juices.

5. DETERMINATION OF ACID MAGENTA—GIRARD'S METHOD.<sup>a</sup>

Add to 100 cc of the solution to be tested 2 cc of potassium hydroxid (5 to 100). If this does not neutralize the acid, add enough to do it. Then add 4 cc of mercuric acetate (10 to 100), agitate and filter. The filtrate should be colorless and slightly alkaline. Acidify with a slight excess of dilute sulphuric acid, and if the solution remains uncolored there is no acid magenta present. If it becomes a light violet-red and there has been no other dye shown by the amyl-alcohol extracts, the presence of acid magenta is shown.

Acid magenta in acid solution dyes wool a magenta red. Wool dyed with it is turned yellow by strong hydrochloric acid, decolorized by ammonium hydroxid, and regains its color when washed with water.

## 6.—TEST FOR MARTIUS YELLOW OR NAPHTHALENE YELLOW.

Extract with 95 per cent alcohol from an acidulated sample. Evaporate the alcoholic solution to dryness with a piece of wool, which will be dyed a bright yellow, and test the dyed wool. Both sulphuric and hydrochloric acids completely decolorize it.

7.—ROTA'S METHOD OF IDENTIFICATION OF ORGANIC COLORING MATTER.<sup>b</sup>

The coloring matters are divided into four groups by the use of stannous chlorid and hydrochloric acid and of caustic potash.

The reagents are a 10 per cent solution of stannous chlorid and a 20 per cent solution of caustic potash.

Dilute the aqueous or alcoholic solution of coloring matter to about 1 to 10,000. This strength is not of vital importance, but the color must not be too deep, as it will mask the reduction in some cases, such as the safranins, where it is slow and not complete. Add to the solution a few drops of stannous chlorid and a few drops of hydrochloric acid; shake, and heat to boiling. Care must be taken to carry along for comparison a solution of the coloring matter acidified with hydrochloric acid, in order not to mistake the action of the acid alone for reduction. Some of the colors—for instance, safranins and indulins—are slow to be reduced and must be allowed to stand for some time. For the stannous chlorid and hydrochloric acid can be substituted a solution of tin in strong hydrochloric acid.

<sup>a</sup>Girard & Dupré, *Analyse des Matières, Alimentaires, etc.*, p. 169; Winton, Conn. Expt. Sta. Rept., 1899, Pt. II, 132.

<sup>b</sup>Chem. Ztg., 1898, 22, 437-442; Analyst, 1899, 24, 41.

As soon as the group is determined it is possible to carry the work further by reference to tables of coloring matter<sup>a</sup> in which the physical, chemical, and tinctorial properties are given; but it is impossible for the published books to keep up with the new dyes which are constantly being discovered, so that the tables are never complete, although they will, as a rule, contain all the data necessary.

*Classification of organic coloring matters.*

[A portion of the aqueous or alcoholic solution is treated with HCl and SnCl<sub>2</sub>.]

Complete decolorization. Reducible coloring matters. Colorless solution is treated with Fe <sub>2</sub> Cl <sub>6</sub> or shaken, with exposure to air.				The color changed no further than with HCl alone. Nonreducible colors. A part of original solution is mixed with 20 per cent KOH and warmed.			
The liquid remains unchanged. Not reoxidizable coloring matters.		The original color restored. Reoxidizable coloring matters.		Decolorization or a precipitate. Imido-carboquinone coloring matters.		No precipitation. Liquid becomes more colored. Oxy-carboquinone coloring matters.	
CLASS I.		CLASS II.		CLASS III.		CLASS IV.	
Nitro, nitroso, and azo colors, including azoxy and hydrazo colors. Picric acid, naphthol yellow, Ponceau, Bordeaux, and Congo red.		Indogenide and imidoquinone coloring matters, methylene blue, safranin, indigo-carmin.		Amido-derivatives of di and triphenylmethane, auramines, acridines, quinolines, and color derivatives of thio benzenil. Fuchsin, rosaniline, auramine.		Nonamide diphenylmethane, oxy-ketone, and most of natural organic coloring matters. Eosines, aurin, alizarin.	

<sup>a</sup>Schultz and Julius, Tabellarische Übersicht der künstlichen organischen Farbstoffe; Allen, Commercial Organic Analysis, 3d ed., Vol. III, pt. 1, pp. 529-565.

• *Characteristics of organic coloring matters.*

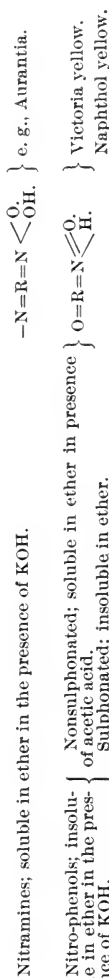
CLASS I.—REDUCED BY  $\text{HCl} + \text{SnCl}_2$  AND NOT REOXIDIZABLE.

*Nitro-coloring matters.*

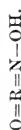


Yellow or orange, soluble in water. Wool and silk dyed directly, but not cotton. The aqueous solution shows tendency to decolorization with  $\text{HCl}$ . With  $\text{HCl} + \text{SnCl}_2$  partially reduced, giving red nitro-amido derivatives (nitramines) or nitro-phenols turning red in  $\text{KOH}$ .

Nitramines; soluble in ether in the presence of  $\text{KOH}$ .



*Nitro-coloring matters.*



Brown or green, usually insoluble in water; indirect for fibers; with  $\text{H}_2\text{SO}_4 + \text{CHOH}$  give blue color (Liebermann's reaction).

Nonsulphonated; insoluble in water; soluble in alcohol; soluble in ether in presence of acetic acid.

Sulphonated; soluble in water; insoluble in ether.

Colored; shaken with dilute acetic acid yields to it the original color. Basic coloring matters.

Nonsulphonated amido-azo coloring matters.

Oxyazo coloring matter without carboxyl.

Oxyazo coloring matters with carboxyl group.

Nonamido compounds unaltered by  $\text{HNO}_3$ .

Amido compounds changed by  $\text{HNO}_3$ .

Colored solution; not yielding its color to dilute acetic acid. Neutral coloring matters.

Nonsulphonated; extracted by ether from dilute solution in acetic acid.

Sulphonated; not extracted by ether from solution in dilute acetic acid.

Colorless solution; yields nothing to acetic acid. Acid coloring matter.

*Azo-coloring matters.*



Their aqueous solution decomposed with  $\text{KOH}$  and extracted with ether gives an ethereal extract with annexed characteristics.

$-\text{N}=\text{R}=\text{N}-\text{NHR}$ .

$\text{O}=\text{R}=\text{N}-\text{HR}$ .

Indirect for cotton wool.

Direct for cotton wool.

Indirect for cotton wool.

Direct for cotton wool.

Indirect for cotton wool.

Direct for cotton wool.

Dioxine (L).

Naphthol green.

Bismarck brown.

Sudan 1 (A).

Diamond yellow (By).

Chrysamine.

Bordeaux B (A).

Azo blue (A).

Solid yellow N (P).

Congo red (A).

*Characteristics of organic coloring matters—Continued.*

CLASS II.—REDUCED BY HCl+SnCl<sub>2</sub> AND REOXIDIZABLE.

<p>The ethereal solution is colored or colorless, and yields the original color to 5 per cent acetic acid. Basic coloring matters fixed on wool in alkaline bath.</p>	<p>The solution is readily reduced by HCl+SnCl<sub>2</sub> in the cold.</p>	<p>Oxyazines (no sulphur).</p>	$\begin{array}{c} \text{R} \\ \diagup \quad \diagdown \\ \text{N} \quad \text{O} \\ \diagdown \quad \diagup \\ \text{R} \end{array} \quad \text{R} \quad \text{N} \equiv \text{N} \equiv \text{N}$	<p>Nile blue A (B).</p>
	<p>The colored solution is reduced but slowly and incompletely even on warming and with the addition of much SnCl<sub>2</sub>+HCl.</p>	<p>Thiazines (sulphur).</p>	$\begin{array}{c} \text{R}_1 \\ \diagup \quad \diagdown \\ \text{N} \quad \text{S} \\ \diagdown \quad \diagup \\ \text{R} \end{array} \quad \text{R} \quad \text{N} \equiv \text{N} \equiv \text{N}$	<p>Methylene blue.</p>
	<p>Blue coloring matters changed by HCl on warming.</p>	<p>Indulines; blue color with conc. H<sub>2</sub>SO<sub>4</sub>. Blue on dilution.</p>	$\begin{array}{c} \text{R}_1 \\ \diagup \quad \diagdown \\ \text{N} \quad \text{N} \\ \diagdown \quad \diagup \\ \text{R} \end{array} \quad \text{R} \quad \text{N} \equiv \text{N} \equiv \text{N}$	<p>Induline soluble in alcohol.</p>
<p>Colored; does not yield the color to acetic acid. Neutral coloring matters. Insoluble in water. Soluble in alcohol. Fixed on fibers in bath.</p>	<p>Red or blue coloring matters. Unaltered by HCl. With HNO<sub>3</sub> yield isatin.</p>	<p>Safranins; green color with H<sub>2</sub>SO<sub>4</sub>. On dilution blue; then violet.</p>	$\begin{array}{c} \text{R} \\ \diagup \quad \diagdown \\ \text{N} \quad \text{N} \\ \diagdown \quad \diagup \\ \text{R} \end{array} \quad \text{R} \quad \text{N} \equiv \text{N} \equiv \text{N}$	<p>Safranin T. extra (A).</p>
	<p>Nonsulphonated. Soluble in ether in presence of acetic acid.</p>	<p>Indophenols.</p>	$\begin{array}{c} \text{R} \\ \diagup \quad \diagdown \\ \text{R} \quad \text{O} \\ \diagdown \quad \diagup \\ \text{R} \end{array}$	<p>Indophenol.</p>
<p>Uncolored; yields nothing to acetic acid. Acid coloring matters. Soluble in water. Fixed on wool in acid bath.</p>	<p>Sulphonated. Insoluble in ether under all circumstances.</p>	<p>Indogenides.</p>	$\begin{array}{c} \text{R} \\ \diagup \quad \diagdown \\ \text{NH} \quad \text{CO} \\ \diagdown \quad \diagup \\ \text{C} \end{array} \quad \text{R} \quad \text{N} \equiv \text{N} \equiv \text{N}$	<p>Indigotin.</p>
	<p>Reduced by SnCl<sub>2</sub>+HCl.</p>	<p>Oxazines.</p>	$\begin{array}{c} \text{R} \\ \diagup \quad \diagdown \\ \text{N} \quad \text{O} \\ \diagdown \quad \diagup \\ \text{R} \end{array} \quad \text{R} \quad \text{N} \equiv \text{N} \equiv \text{N}$	<p>Fluorescent blue; orcein.</p>
<p>The aqueous or alcoholic solution is treated with KOH and extracted with ether.</p>	<p>Not reduced by SnCl<sub>2</sub>+HCl.</p>	<p>Sulphonated indogenides. Sulphonated thiazines.</p>	<p>Sulphonated indogenides. Sulphonated thiazines.</p>	<p>Indigo carmine. Thio Carmine R (C). Soluble nigrosin.</p>

The ethereal solution washed with water has the annexed characteristics.



*Characteristics of organic coloring matters—Continued.*

CLASS IV.—COLORING MATTERS NOT REDUCED BY  $\text{SnCl}_2 + \text{HCl}$ . CONTAINING THE OXY-QUINONE CARBON CHROMOPHORE  $\text{O}=\text{R}=\text{C}=\text{}$ .

<p>The alcoholic solution of the coloring matter treated with a few drops of a dilute solution of <math>\text{FeCl}_3</math> (1:1000).</p>	<p>Remains unaltered. Nonamido triphenyl- methane coloring matters. Usually soluble in water and direct for wool.</p>	<p>The coloring matter is dissolved or suspended in boiling water.</p>	<p>Not directly fixed on wool. Most of them in- soluble in water. Soluble in alcohol without fluorescence.</p>	<p>Aurins.</p>	$\begin{array}{c} \text{R}^1 \\ \diagup \\ \text{C}=\text{R}^1 \\ \diagdown \\ \text{R}=\text{O} \end{array}$	<p>Aurin.</p>
	<p>Changes to green or olive green.</p>	<p>Dissolves with yellow or reddish yellow color. Monoketones.</p>	<p>Fixed directly on wool. Most of them soluble in water and alcohol. Fluorescent.</p>	<p>Phthaléins.</p>	$\begin{array}{c} \text{R}^1 \\ \diagup \\ \text{C}=\text{R}^1 \\ \diagdown \\ \text{R}=\text{O} \end{array}$	<p>Eosin.</p>
<p>Oxy-ketone coloring matters. Most of them insoluble in water and indirect for fibers.</p>	<p>The original coloring matter is treated with faintly alkaline water is (KOH, 1 per cent).</p>	<p>The alkaline so- lution treated with excess of HCl.</p>	<p>Inclined to decolorization, especially on warming (with decomposition).</p>	<p>Benzophenones.</p>	$\begin{array}{c} \text{R} \\ \diagup \\ \text{CO} \\ \diagdown \\ \text{CO} \end{array}$	<p>Alizarin yellow A (B).</p>
			<p>Colored intense yellow with- out decomposition.</p>	<p>Flavones.</p>	$\begin{array}{c} \text{R} \\ \diagup \\ \text{CO} \\ \diagdown \\ \text{CO} \end{array}$	<p>Quercetin.</p>
		<p>The alkaline acid- solution with acetic acid.</p>	<p>The free coloring matter pre- cipitated. Usually soluble in ether and indirect for fibers.</p>	<p>Nonsulphonated anthraquinones.</p>	$\begin{array}{c} \text{R} \\ \diagup \\ \text{CO} \\ \diagdown \\ \text{CO} \end{array}$	<p>Alizarin.</p>
		<p>Dissolves with red, reddish violet, green, or blue color. Diketones (quinones).</p>	<p>Coloring matters remain in solution. Insoluble in ether fixed directly on wool.</p>	<p>Sulphonated an- thraquinones.</p>	$\begin{array}{c} \text{R} \\ \diagup \\ \text{CO} \\ \diagdown \\ \text{CO} \end{array}$	<p>Sulphonated ali- zarin (alizarin red).</p>

## 8.—DETERMINATION OF VEGETABLE COLORS.

A great many tests for vegetable colors are given, depending largely on color reactions with different reagents, but these must be used with very great discrimination, as they depend very largely on a fine judgment of shades of colors which many eyes are not able to distinguish.

A great deal of work has been done on detection of vegetable colors,<sup>a</sup> but only in a very few cases are the reactions specific enough to be decisive.

9.—DETECTION OF TURMERIC.<sup>b</sup>

Extract the color with alcohol. Dip a piece of filter paper into this tincture and dry at 100° C.

Then moisten in a weak solution of boric acid to which a few drops of hydrochloric acid have been added. On drying this, a cherry red color will be developed in the presence of turmeric which is characteristic.

## 10.—DETECTION OF CARAMEL.

AMTIIOR TEST.<sup>c</sup>

Ten cubic centimeters of the solution to be tested are put into a high, narrow glass with perpendicular sides, as, for example, a small bottle; add from 30 to 50 cc of paraldehyde, depending on the intensity of the coloring, and enough absolute alcohol to make the solutions mix. In the presence of caramel a brownish yellow to dark-brown precipitate will collect in the bottom of the glass. Decant the liquor, wash once with absolute alcohol, dissolve in small amount of hot water, and filter. The color of this will give some idea as to the amount of caramel present.

It is not allowable to concentrate a solution by evaporation on a steam bath, as caramel may be formed; if it is necessary to concentrate it must be done over sulphuric acid or at diminished pressure.

In order to further identify the color it is poured into a freshly prepared solution of phenylhydrazin (2 parts phenylhydrazin-hydrochlorid, 3 parts sodium acetate, and 20 parts of water). The presence of a considerable quantity of caramel gives a dark-brown precipitate in the cold, which is hastened by heating a little.

In the case of a very small amount it takes some hours for it to collect.

## 11.—DETECTION OF COCHINEAL.

Cochineal is used to a certain extent as a coloring matter in foods, and a very satisfactory test for it is that given in Girard and Dupré.<sup>d</sup> Dissolve the food product in water, filtering if necessary. Acidulate with hydrochloric acid and extract with amyl alcohol, which becomes colored more or less yellow or orange, depending on the quantity of cochineal present. Separate the amyl alcohol and wash until neutral. Then separate into two portions; to the first add drop by drop a very dilute solution of uranium acetate, shaking thoroughly after each addition. In the presence of cochineal a characteristic emerald-green color is produced.<sup>e</sup>

To the second portion add a drop or so of ammonia, and in presence of cochineal a violet coloration results. This, however, is not so sensitive to very small amounts as the first tests, and many fruit colors give tests hardly to be distinguished.

Cochineal carmine is liable to contain tin, as it is often a tin lake, although alum is also used. It is also liable to adulteration with lead compounds.

<sup>a</sup> Girard and Dupré, *Analyse des Matières Alimentaires*, etc., 580-581, also 169; A. W. Blythe, *Foods*, their Comp. and Anal., p. 91-109; Allen Com. Org. Anal., Vol. III, Pt. I; E. Brucher, *Fals. Subst. Alim.*, p. 162; W. Lenz, *Ztschr. anal. Chem.*, 1885, **24**, 285.

<sup>b</sup> Allen, *Com. Org. Anal.*, Vol. III, Pt. I, p. 359; U. S. Dept. of Agr., Div. of Chem., Bul. 51, p. 131.

<sup>c</sup> *Ztsch. anal. Chem.*, 1885, **24**, 30; Borgemann, *Anal. des Weines*, p. 98.

<sup>d</sup> *Analyse des Matières Alimentaires*, etc., p. 580.

<sup>e</sup> The writer has tested this reaction on a number of amyl alcohol extracts from fruits, and in no case was there any chance of mistake in the reaction. Most fruits give a brown color, while blackberries and currants give a bluish color.



## REFERENCE TABLES.

TABLE I.—*Specific gravity and percentage of alcohol.*

[According to Squibb.]

Per cent alcohol by volume.	Specific gravity.		Per cent alcohol by volume.	Specific gravity.		Per cent alcohol by volume.	Specific gravity.	
	At 15.56° C.	At 25° C.		At 15.56° C.	At 25° C.		At 15.56° C.	At 25° C.
1	.9985	.9970	36	.9578	.9521	71	.8875	.8796
2	.9970	.9953	37	.9565	.9507	72	.8850	.8771
3	.9956	.9938	38	.9550	.9489	73	.8825	.8746
4	.9942	.9922	39	.9535	.9473	74	.8799	.8719
5	.9930	.9909	40	.9519	.9456	75	.8769	.8689
6	.9914	.9893	41	.9503	.9438	76	.8745	.8665
7	.9898	.9876	42	.9490	.9424	77	.8721	.8641
8	.9890	.9868	43	.9470	.9402	78	.8696	.8616
9	.9878	.9855	44	.9452	.9382	79	.8664	.8583
10	.9869	.9846	45	.9434	.9363	80	.8639	.8558
11	.9855	.9831	46	.9416	.9343	81	.8611	.8530
12	.9841	.9816	47	.9396	.9323	82	.8581	.8500
13	.9828	.9801	48	.9381	.9307	83	.8557	.8476
14	.9821	.9793	49	.9362	.9288	84	.8526	.8444
15	.9815	.9787	50	.9343	.9267	85	.8496	.8414
16	.9802	.9773	51	.9323	.9246	86	.8466	.8384
17	.9789	.9759	52	.9303	.9226	87	.8434	.8352
18	.9778	.9746	53	.9283	.9205	88	.8408	.8326
19	.9766	.9733	54	.9262	.9184	89	.8373	.8291
20	.9760	.9726	55	.9242	.9164	90	.8340	.8258
21	.9753	.9719	56	.9221	.9143	91	.8305	.8223
22	.9741	.9706	57	.9200	.9122	92	.8272	.8191
23	.9728	.9692	58	.9178	.9100	93	.8237	.8156
24	.9716	.9678	59	.9160	.9081	94	.8199	.8118
25	.9709	.9668	60	.9135	.9056	95	.8164	.8083
26	.9698	.9655	61	.9113	.9034	96	.8125	.8044
27	.9691	.9646	62	.9090	.9011	97	.8084	.8003
28	.9678	.9631	63	.9069	.8989	98	.8041	.7960
29	.9665	.9617	64	.9047	.8969	99	.7995	.7914
30	.9652	.9603	65	.9025	.8947	100	.7946	.7865
31	.9643	.9594	66	.9001	.8923			
32	.9631	.9582	67	.8973	.8895			
33	.9618	.9567	68	.8949	.8870			
34	.9609	.9556	69	.8925	.8846			
35	.9593	.9538	70	.8900	.8821			

TABLE II.—*Percentage of alcohol.*

[Recalculated from the determinations of Gilpin, Drinkwater, and Squibb.]

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
1.00000	0.00	0.00	0.00	.99884	0.75	0.60	0.60	0.99775	1.50	1.19	1.19
.99992	0.05	0.04	0.04	.99877	0.80	0.64	0.64	.99768	1.55	1.23	1.23
.99984	0.10	0.08	0.08	.99869	0.85	0.67	0.67	.99760	1.60	1.27	1.27
.99976	0.15	0.12	0.12	.99861	0.90	0.71	0.71	.99753	1.65	1.31	1.31
.99968	0.20	0.16	0.16	.99854	0.95	0.75	0.75	.99745	1.70	1.35	1.35
.99961	0.25	0.20	0.20	.99849	1.00	0.79	0.79	.99738	1.75	1.39	1.39
.99953	0.30	0.24	0.24	.99842	1.05	0.83	0.83	.99731	1.80	1.43	1.43
.99945	0.35	0.28	0.28	.99834	1.10	0.87	0.87	.99723	1.85	1.47	1.47
.99937	0.40	0.32	0.32	.99827	1.15	0.91	0.91	.99716	1.90	1.51	1.51
.99930	0.45	0.36	0.36	.99819	1.20	0.95	0.95	.99708	1.95	1.55	1.55
.99923	0.50	0.40	0.40	.99812	1.25	0.99	0.99	.99701	2.00	1.59	1.59
.99915	0.55	0.44	0.44	.99805	1.30	1.03	1.03	.99694	2.05	1.63	1.62
.99907	0.60	0.48	0.48	.99797	1.35	1.07	1.07	.99687	2.10	1.67	1.66
.99900	0.65	0.52	0.52	.99790	1.40	1.11	1.11	.99679	2.15	1.71	1.70
.99892	0.70	0.56	0.56	.99782	1.45	1.15	1.15	.99672	2.20	1.75	1.74

TABLE II.—*Percentage of alcohol*—Continued.

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
0.99665	2.25	1.79	1.78	0.99215	5.50	4.40	4.37	0.98807	8.75	7.03	6.95
.99658	2.30	1.83	1.82	.99208	5.55	4.44	4.40	.98801	8.80	7.07	6.99
.99651	2.35	1.87	1.86	.99202	5.60	4.48	4.44	.98795	8.85	7.11	7.03
.99643	2.40	1.91	1.90	.99195	5.65	4.52	4.48	.98789	8.90	7.15	7.07
.99636	2.45	1.95	1.94	.99189	5.70	4.56	4.52	.98783	8.95	7.19	7.11
.99629	2.50	1.99	1.98	.99182	5.75	4.60	4.56	.98777	9.00	7.23	7.14
.99622	2.55	2.03	2.02	.99175	5.80	4.64	4.60	.98771	9.05	7.27	7.18
.99615	2.60	2.07	2.06	.99169	5.85	4.68	4.64	.98765	9.10	7.31	7.22
.99607	2.65	2.11	2.10	.99162	5.90	4.72	4.68	.98759	9.15	7.35	7.26
.99600	2.70	2.15	2.14	.99156	5.95	4.76	4.72	.98754	9.20	7.39	7.30
.99593	2.75	2.19	2.18	.99149	6.00	4.80	4.76	.98748	9.25	7.43	7.34
.99586	2.80	2.23	2.22	.99143	6.05	4.84	4.80	.98742	9.30	7.48	7.38
.99579	2.85	2.27	2.26	.99136	6.10	4.88	4.84	.98736	9.35	7.52	7.42
.99571	2.90	2.31	2.30	.99130	6.15	4.92	4.88	.98730	9.40	7.56	7.46
.99564	2.95	2.35	2.34	.99123	6.20	4.96	4.92	.98724	9.45	7.60	7.50
.99557	3.00	2.39	2.38	.99117	6.25	5.	4.96	.98719	9.50	7.64	7.54
.99550	3.05	2.43	2.42	.99111	6.30	5.05	5.00	.98713	9.55	7.68	7.58
.99543	3.10	2.47	2.46	.99104	6.35	5.09	5.04	.98707	9.60	7.72	7.62
.99536	3.15	2.51	2.50	.99098	6.40	5.13	5.08	.98701	9.65	7.76	7.66
.99529	3.20	2.55	2.54	.99091	6.45	5.17	5.12	.98695	9.70	7.80	7.70
.99522	3.25	2.59	2.58	.99085	6.50	5.21	5.16	.98689	9.75	7.84	7.74
.99515	3.30	2.64	2.62	.99079	6.55	5.25	5.20	.98683	9.80	7.88	7.78
.99508	3.35	2.68	2.66	.99072	6.60	5.29	5.24	.98678	9.85	7.92	7.82
.99501	3.40	2.72	2.70	.99066	6.65	5.33	5.28	.98672	9.90	7.96	7.85
.99494	3.45	2.76	2.74	.99059	6.70	5.37	5.32	.98666	9.95	8.00	7.89
.99487	3.50	2.80	2.78	.99053	6.75	5.41	5.36	.98660	10.00	8.04	7.93
.99480	3.55	2.84	2.82	.99047	6.80	5.45	5.40	.98654	10.05	8.08	7.97
.99473	3.60	2.88	2.86	.99040	6.85	5.49	5.44	.98649	10.10	8.12	8.01
.99466	3.65	2.92	2.90	.99034	6.90	5.53	5.48	.98643	10.15	8.16	8.05
.99459	3.70	2.96	2.94	.99027	6.95	5.57	5.52	.98637	10.20	8.20	8.09
.99452	3.75	3.00	2.98	.99021	7.00	5.61	5.56	.98632	10.25	8.24	8.13
.99445	3.80	3.04	3.02	.99015	7.05	5.65	5.60	.98626	10.30	8.29	8.17
.99438	3.85	3.08	3.06	.99009	7.10	5.69	5.64	.98620	10.35	8.33	8.21
.99431	3.90	3.12	3.10	.99002	7.15	5.73	5.68	.98614	10.40	8.37	8.25
.99424	3.95	3.16	3.14	.98996	7.20	5.77	5.72	.98609	10.45	8.41	8.29
.99417	4.00	3.20	3.18	.98990	7.25	5.81	5.76	.98603	10.50	8.45	8.33
.99410	4.05	3.24	3.22	.98984	7.30	5.86	5.80	.98597	10.55	8.49	8.37
.99403	4.10	3.28	3.26	.98978	7.35	5.90	5.84	.98592	10.60	8.53	8.41
.99397	4.15	3.32	3.30	.98971	7.40	5.94	5.88	.98586	10.65	8.57	8.45
.99390	4.20	3.36	3.34	.98965	7.45	5.98	5.92	.98580	10.70	8.61	8.49
.99383	4.25	3.40	3.38	.98959	7.50	6.02	5.96	.98575	10.75	8.65	8.53
.99376	4.30	3.44	3.42	.98953	7.55	6.06	6.00	.98569	10.80	8.70	8.57
.99369	4.35	3.48	3.46	.98947	7.60	6.10	6.04	.98563	10.85	8.74	8.61
.99363	4.40	3.52	3.50	.98940	7.65	6.14	6.07	.98557	10.90	8.78	8.65
.99356	4.45	3.56	3.54	.98934	7.70	6.18	6.11	.98552	10.95	8.82	8.69
.99349	4.50	3.60	3.58	.98928	7.75	6.22	6.15	.98546	11.00	8.86	8.73
.99342	4.55	3.64	3.62	.98922	7.80	6.26	6.19	.98540	11.05	8.90	8.77
.99335	4.60	3.68	3.66	.98916	7.85	6.30	6.23	.98535	11.10	8.94	8.81
.99329	4.65	3.72	3.70	.98909	7.90	6.34	6.27	.98529	11.15	8.98	8.85
.99322	4.70	3.76	3.74	.98903	7.95	6.38	6.31	.98524	11.20	9.02	8.89
.99315	4.75	3.80	3.77	.98897	8.00	6.42	6.35	.98518	11.25	9.07	8.93
.99308	4.80	3.84	3.81	.98891	8.05	6.46	6.39	.98513	11.30	9.11	8.97
.99301	4.85	3.88	3.85	.98885	8.10	6.50	6.43	.98507	11.35	9.15	9.01
.99295	4.90	3.92	3.89	.98879	8.15	6.54	6.47	.98502	11.40	9.19	9.05
.99288	4.95	3.96	3.93	.98873	8.20	6.58	6.51	.98496	11.45	9.23	9.09
.99281	5.00	4.00	3.97	.98867	8.25	6.62	6.55	.98491	11.50	9.27	9.13
.99274	5.05	4.04	4.01	.98861	8.30	6.67	6.59	.98485	11.55	9.31	9.17
.99268	5.10	4.08	4.05	.98855	8.35	6.71	6.63	.98479	11.60	9.35	9.21
.99261	5.15	4.12	4.09	.98849	8.40	6.75	6.67	.98474	11.65	9.39	9.25
.99255	5.20	4.16	4.13	.98843	8.45	6.79	6.71	.98468	11.70	9.43	9.29
.99248	5.25	4.20	4.17	.98837	8.50	6.83	6.75	.98463	11.75	9.47	9.32
.99241	5.30	4.24	4.21	.98831	8.55	6.87	6.79	.98457	11.80	9.51	9.36
.99235	5.35	4.28	4.25	.98825	8.60	6.91	6.83	.98452	11.85	9.55	9.40
.99228	5.40	4.32	4.29	.98819	8.65	6.95	6.87	.98446	11.90	9.59	9.44
.99222	5.45	4.36	4.33	.98813	8.70	6.99	6.91	.98441	11.95	9.63	9.48

TABLE II.—*Percentage of alcohol*—Continued.

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
.98435	12.00	9.67	9.52	.98088	15.25	12.33	12.10	.97758	18.50	15.02	14.68
.98430	12.05	9.71	9.56	.98083	15.30	12.38	12.14	.97753	18.55	15.06	14.72
.98424	12.10	9.75	9.60	.98078	15.35	12.42	12.18	.97748	18.60	15.10	14.76
.98419	12.15	9.79	9.64	.98073	15.40	12.46	12.22	.97743	18.65	15.14	14.80
.98413	12.20	9.83	9.68	.98068	15.45	12.50	12.26	.97738	18.70	15.18	14.84
.98408	12.25	9.87	9.72	.98063	15.50	12.54	12.30	.97733	18.75	15.22	14.88
.98402	12.30	9.92	9.76	.98057	15.55	12.58	12.34	.97728	18.80	15.27	14.92
.98397	12.35	9.96	9.80	.98052	15.60	12.62	12.37	.97723	18.85	15.31	14.96
.98391	12.40	10.00	9.84	.98047	15.65	12.66	12.41	.97718	18.90	15.38	15.00
.98386	12.45	10.04	9.88	.98042	15.70	12.70	12.45	.97713	18.95	15.39	15.04
.98381	12.50	10.08	9.92	.98037	15.75	12.75	12.49	.97708	19.00	15.43	15.08
.98375	12.55	10.12	9.96	.98032	15.80	12.79	12.53	.97703	19.05	15.47	15.11
.98370	12.60	10.16	10.00	.98026	15.85	12.83	12.57	.97698	19.10	15.51	15.15
.98364	12.65	10.20	10.03	.98021	15.90	12.87	12.61	.97693	19.15	15.55	15.19
.98359	12.70	10.24	10.07	.98016	15.95	12.91	12.65	.97688	19.20	15.59	15.23
.98353	12.75	10.28	10.11	.98011	16.00	12.95	12.69	.97683	19.25	15.63	15.27
.98348	12.80	10.33	10.15	.98005	16.05	12.99	12.73	.97678	19.30	15.68	15.31
.98342	12.85	10.37	10.19	.98000	16.10	13.03	12.77	.97673	19.35	15.72	15.35
.98337	12.90	10.41	10.23	.97996	16.15	13.08	12.81	.97668	19.40	15.76	15.39
.98331	12.95	10.45	10.27	.97991	16.20	13.12	12.85	.97663	19.45	15.80	15.43
.98326	13.00	10.49	10.31	.97986	16.25	13.16	12.89	.97658	19.50	15.84	15.47
.98321	13.05	10.53	10.35	.97980	16.30	13.20	12.93	.97653	19.55	15.88	15.51
.98315	13.10	10.57	10.39	.97975	16.35	13.24	12.97	.97648	19.60	15.93	15.55
.98310	13.15	10.61	10.43	.97970	16.40	13.29	13.01	.97643	19.65	15.97	15.59
.98305	13.20	10.65	10.47	.97965	16.45	13.33	13.05	.97638	19.70	16.01	15.63
.98299	13.25	10.69	10.51	.97960	16.50	13.37	13.09	.97633	19.75	16.05	15.67
.98294	13.30	10.74	10.55	.97955	16.55	13.41	13.13	.97628	19.80	16.09	15.71
.98289	13.35	10.78	10.59	.97950	16.60	13.45	13.17	.97623	19.85	16.14	15.75
.98283	13.40	10.82	10.63	.97945	16.65	13.49	13.21	.97618	19.90	16.18	15.79
.98278	13.45	10.86	10.67	.97940	16.70	13.53	13.25	.97613	19.95	16.22	15.83
.98273	13.50	10.90	10.71	.97935	16.75	13.57	13.29	.97608	20.00	16.26	15.87
.98267	13.55	10.94	10.75	.97929	16.80	13.62	13.33	.97603	20.05	16.30	15.91
.98262	13.60	10.98	10.79	.97924	16.85	13.66	13.37	.97598	20.10	16.34	15.95
.98256	13.65	11.02	10.83	.97919	16.90	13.70	13.41	.97593	20.15	16.38	15.99
.98251	13.70	11.06	10.87	.97914	16.95	13.74	13.45	.97588	20.20	16.42	16.03
.98246	13.75	11.11	10.91	.97909	17.00	13.78	13.49	.97583	20.25	16.46	16.06
.98240	13.80	11.15	10.95	.97904	17.05	13.82	13.53	.97578	20.30	16.51	16.10
.98235	13.85	11.19	10.99	.97899	17.10	13.86	13.57	.97573	20.35	16.56	16.14
.98230	13.90	11.23	11.03	.97894	17.15	13.90	13.61	.97568	20.40	16.59	16.18
.98224	13.95	11.27	11.07	.97889	17.20	13.94	13.65	.97563	20.45	16.63	16.22
.98219	14.00	11.31	11.11	.97884	17.25	13.98	13.69	.97558	20.50	16.67	16.26
.98214	14.05	11.35	11.15	.97879	17.30	14.03	13.73	.97553	20.55	16.71	16.30
.98209	14.10	11.39	11.19	.97874	17.35	14.07	13.77	.97548	20.60	16.75	16.34
.98203	14.15	11.43	11.23	.97869	17.40	14.11	13.81	.97543	20.65	16.80	16.38
.98198	14.20	11.47	11.27	.97864	17.45	14.15	13.85	.97538	20.70	16.84	16.42
.98193	14.25	11.52	11.31	.97859	17.50	14.19	13.89	.97533	20.75	16.88	16.46
.98188	14.30	11.56	11.35	.97853	17.55	14.23	13.92	.97528	20.80	16.92	16.50
.98182	14.35	11.60	11.39	.97848	17.60	14.27	13.96	.97523	20.85	16.96	16.54
.98177	14.40	11.64	11.43	.97843	17.65	14.31	14.00	.97517	20.90	17.01	16.58
.98172	14.45	11.68	11.47	.97838	17.70	14.35	14.04	.97512	20.95	17.05	16.62
.98167	14.50	11.72	11.51	.97833	17.75	14.40	14.08	.97507	21.00	17.09	16.66
.98161	14.55	11.76	11.55	.97828	17.80	14.44	14.12	.97502	21.05	17.13	16.70
.98156	14.60	11.80	11.59	.97823	17.85	14.48	14.16	.97497	21.10	17.17	16.74
.98151	14.65	11.84	11.63	.97818	17.90	14.52	14.20	.97492	21.15	17.22	16.78
.98146	14.70	11.88	11.67	.97813	17.95	14.56	14.24	.97487	21.20	17.26	16.82
.98140	14.75	11.93	11.71	.97808	18.00	14.60	14.28	.97482	21.25	17.30	16.86
.98135	14.80	11.97	11.75	.97803	18.05	14.64	14.32	.97477	21.30	17.34	16.90
.98130	14.85	12.01	11.79	.97798	18.10	14.68	14.36	.97472	21.35	17.38	16.94
.98125	14.90	12.05	11.82	.97793	18.15	14.73	14.40	.97467	21.40	17.43	16.98
.98119	14.95	12.09	11.86	.97788	18.20	14.77	14.44	.97462	21.45	17.47	17.02
.98114	15.00	12.13	11.90	.97783	18.25	14.81	14.48	.97457	21.50	17.51	17.06
.98108	15.05	12.17	11.94	.97778	18.30	14.85	14.52	.97451	21.55	17.55	17.10
.98104	15.10	12.21	11.98	.97773	18.35	14.89	14.56	.97446	21.60	17.59	17.14
.98099	15.15	12.25	12.02	.97768	18.40	14.94	14.60	.97441	21.65	17.63	17.18
.98093	15.20	12.29	12.06	.97763	18.45	14.98	14.64	.97436	21.70	17.67	17.22

TABLE II.—*Percentage of alcohol*—Continued.

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
0.97431	21.75	17.71	17.26	0.97097	25.00	20.43	19.84	0.96744	28.25	23.17	22.42
.97426	21.80	17.76	17.30	.97092	25.05	20.47	19.88	.96738	28.30	23.21	22.45
.97421	21.85	17.80	17.34	.97086	25.10	20.51	19.92	.96732	28.35	23.25	22.49
.97416	21.90	17.84	17.38	.97081	25.15	20.56	19.96	.96726	28.40	23.30	22.53
.97411	21.95	17.88	17.42	.97076	25.20	20.60	20.00	.96721	28.45	23.34	22.57
.97406	22.00	17.92	17.46	.97071	25.25	20.64	20.04	.96715	28.50	23.38	22.61
.97401	22.05	17.96	17.50	.97065	25.30	20.68	20.08	.96709	28.55	23.42	22.65
.97396	22.10	18.00	17.54	.97060	25.35	20.72	20.12	.96704	28.60	23.47	22.69
.97391	22.15	18.05	17.58	.97055	25.40	20.77	20.16	.96698	28.65	23.51	22.73
.97386	22.20	18.09	17.62	.97049	25.45	20.81	20.20	.96692	28.70	23.55	22.77
.97381	22.25	18.13	17.66	.97044	25.50	20.85	20.24	.96687	28.75	23.60	22.81
.97375	22.30	18.17	17.70	.97039	25.55	20.89	20.28	.96681	28.80	23.64	22.85
.97370	22.35	18.21	17.74	.97033	25.60	20.93	20.32	.96675	28.85	23.68	22.89
.97365	22.40	18.26	17.78	.97028	25.65	20.98	20.36	.96669	28.90	23.72	22.93
.97360	22.45	18.30	17.82	.97023	25.70	21.02	20.40	.96664	28.95	23.77	22.97
.97355	22.50	18.34	17.86	.97018	25.75	21.06	20.44	.96658	29.00	23.81	23.01
.97350	22.55	18.38	17.90	.97012	25.80	21.10	20.47	.96652	29.05	23.85	23.05
.97345	22.60	18.42	17.94	.97007	25.85	21.14	20.51	.96646	29.10	23.89	23.09
.97340	22.65	18.47	17.98	.97001	25.90	21.19	20.55	.96640	29.15	23.94	23.13
.97335	22.70	18.51	18.02	.96996	25.95	21.23	20.59	.96635	29.20	23.98	23.17
.97330	22.75	18.55	18.06	.96991	26.00	21.27	20.63	.96629	29.25	24.02	23.21
.97324	22.80	18.59	18.10	.96986	26.05	21.31	20.67	.96623	29.30	24.06	23.25
.97319	22.85	18.63	18.14	.96980	26.10	21.35	20.71	.96617	29.35	24.10	23.29
.97314	22.90	18.68	18.18	.96975	26.15	21.40	20.75	.96611	29.40	24.15	23.33
.97309	22.95	18.72	18.22	.96969	26.20	21.44	20.79	.96605	29.45	24.19	23.37
.97304	23.00	18.76	18.26	.96964	26.25	21.48	20.83	.96600	29.50	24.23	23.41
.97299	23.05	18.80	18.29	.96959	26.30	21.52	20.87	.96594	29.55	24.27	23.45
.97294	23.10	18.84	18.33	.96953	26.35	21.56	20.91	.96587	29.60	24.32	23.49
.97289	23.15	18.88	18.37	.96949	26.40	21.61	20.95	.96582	29.65	24.36	23.53
.97283	23.20	18.92	18.41	.96942	26.45	21.65	20.99	.96576	29.70	24.40	23.57
.97278	23.25	18.96	18.45	.96937	26.50	21.69	21.03	.96570	29.75	24.45	23.61
.97273	23.30	19.01	18.49	.96932	26.55	21.73	21.07	.96564	29.80	24.49	23.65
.97268	23.35	19.05	18.53	.96926	26.60	21.77	21.11	.96559	29.85	24.53	23.69
.97263	23.40	19.09	18.57	.96921	26.65	21.82	21.15	.96553	29.90	24.57	23.73
.97258	23.45	19.13	18.61	.96915	26.70	21.86	21.19	.96547	29.95	24.62	23.77
.97253	23.50	19.17	18.65	.96910	26.75	21.90	21.23	.96541	30.00	24.66	23.81
.97247	23.55	19.21	18.69	.96905	26.80	21.94	21.27	.96535	30.05	24.70	23.85
.97242	23.60	19.25	18.73	.96899	26.85	21.98	21.31	.96529	30.10	24.74	23.89
.97237	23.65	19.30	18.77	.96894	26.90	22.03	21.35	.96523	30.15	24.79	23.93
.97232	23.70	19.34	18.81	.96888	26.95	22.07	21.39	.96517	30.20	24.83	23.97
.97227	23.75	19.38	18.84	.96883	27.00	22.11	21.43	.96511	30.25	24.87	24.01
.97222	23.80	19.42	18.88	.96877	27.05	22.15	21.47	.96505	30.30	24.91	24.04
.97216	23.85	19.46	18.92	.96872	27.10	22.20	21.51	.96499	30.35	24.95	24.08
.97211	23.90	19.51	18.96	.96866	27.15	22.24	21.55	.96493	30.40	25.00	24.12
.97206	23.95	19.55	19.00	.96861	27.20	22.28	21.59	.96487	30.45	25.04	24.16
.97201	24.00	19.59	19.04	.96855	27.25	22.33	21.63	.96481	30.50	25.08	24.20
.97196	24.05	19.63	19.08	.96850	27.30	22.37	21.67	.96475	30.55	25.12	24.24
.97191	24.10	19.67	19.12	.96844	27.35	22.41	21.71	.96469	30.60	25.17	24.28
.97185	24.15	19.72	19.16	.96839	27.40	22.45	21.75	.96463	30.65	25.21	24.32
.97180	24.20	19.76	19.20	.96833	27.45	22.50	21.79	.96457	30.70	25.25	24.36
.97175	24.25	19.80	19.24	.96828	27.50	22.54	21.83	.96451	30.75	25.30	24.40
.97170	24.30	19.84	19.28	.96822	27.55	22.58	21.86	.96445	30.80	25.34	24.44
.97165	24.35	19.88	19.32	.96816	27.60	22.62	21.90	.96439	30.85	25.38	24.48
.97159	24.40	19.93	19.36	.96811	27.65	22.67	21.94	.96433	30.90	25.42	24.52
.97154	24.45	19.97	19.40	.96805	27.70	22.71	21.98	.96427	30.95	25.47	24.56
.97149	24.50	20.01	19.44	.96800	27.75	22.75	22.02	.96421	31.00	25.51	24.60
.97144	24.55	20.05	19.48	.96794	27.80	22.79	22.06	.96415	31.05	25.55	24.64
.97139	24.60	20.09	19.52	.96789	27.85	22.83	22.10	.96409	31.10	25.60	24.68
.97133	24.65	20.14	19.56	.96783	27.90	22.88	22.14	.96403	31.15	25.64	24.72
.97128	24.70	20.18	19.60	.96778	27.95	22.92	22.18	.96396	31.20	25.68	24.76
.97123	24.75	20.22	19.64	.96772	28.00	22.96	22.22	.96390	31.25	25.73	24.80
.97118	24.80	20.26	19.68	.96766	28.05	23.00	22.26	.96384	31.30	25.77	24.84
.97113	24.85	20.30	19.72	.96761	28.10	23.04	22.30	.96378	31.35	25.81	24.88
.97107	24.90	20.35	19.76	.96755	28.15	23.09	22.34	.96372	31.40	25.85	24.92
.97102	24.95	20.39	19.80	.96749	28.20	23.13	22.38	.96366	31.45	25.90	24.96

TABLE II.—Percentage of alcohol—Continued.

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
0.96360	31.50	25.94	25.00	0.95943	34.75	28.74	27.58	0.95487	38.00	31.58	30.16
.96353	31.55	25.98	25.04	.95937	34.80	28.78	27.62	.95480	38.05	31.63	30.20
.96347	31.60	26.03	25.08	.95930	34.85	28.83	27.66	.95472	38.10	31.67	30.24
.96341	31.65	26.07	25.12	.95923	34.90	28.87	27.70	.95465	38.15	31.72	30.28
.96335	31.70	26.11	25.16	.95917	34.95	28.92	27.74	.95457	38.20	31.76	30.32
.96329	31.75	26.16	25.20	.95910	35.00	28.96	27.78	.95450	38.25	31.81	30.36
.96323	31.80	26.20	25.24	.95903	35.05	29.00	27.82	.95442	38.30	31.85	30.40
.96316	31.85	26.24	25.28	.95896	35.10	29.05	27.86	.95435	38.35	31.90	30.44
.96310	31.90	26.28	25.32	.95889	35.15	29.09	27.90	.95427	38.40	31.94	30.48
.96304	31.95	26.33	25.36	.95883	35.20	29.13	27.94	.95420	38.45	31.99	30.52
.96298	32.00	26.37	25.40	.95876	35.25	29.18	27.98	.95413	38.50	32.03	30.56
.96292	32.05	26.41	25.44	.95869	35.30	29.22	28.02	.95405	38.55	32.07	30.60
.96285	32.10	26.46	25.48	.95862	35.35	29.26	28.05	.95398	38.60	32.12	30.64
.96279	32.15	26.50	25.52	.95855	35.40	29.30	28.09	.95390	38.65	32.16	30.68
.96273	32.20	26.54	25.56	.95848	35.45	29.35	28.13	.95383	38.70	32.20	30.72
.96267	32.25	26.59	25.60	.95842	35.50	29.40	28.17	.95375	38.75	32.25	30.76
.96260	32.30	26.63	25.64	.95835	35.55	29.43	28.21	.95368	38.80	32.29	30.79
.96254	32.35	26.67	25.68	.95828	35.60	29.48	28.25	.95360	38.85	32.33	30.83
.96248	32.40	26.71	25.71	.95821	35.65	29.52	28.29	.95353	38.90	32.37	30.87
.96241	32.45	26.76	25.75	.95814	35.70	29.57	28.33	.95345	38.95	32.42	30.91
.96235	32.50	26.80	25.79	.95807	35.75	29.61	28.37	.95338	39.00	32.46	30.95
.96229	32.55	26.84	25.83	.95800	35.80	29.65	28.41	.95330	39.05	32.50	30.99
.96222	32.60	26.89	25.87	.95794	35.85	29.70	28.45	.95323	39.10	32.55	31.03
.96216	32.65	26.93	25.91	.95787	35.90	29.74	28.49	.95315	39.15	32.59	31.07
.96210	32.70	26.97	25.95	.95780	35.95	29.79	28.53	.95307	39.20	32.64	31.11
.96204	32.75	27.02	25.99	.95773	36.00	29.83	28.57	.95300	39.25	32.68	31.14
.96197	32.80	27.06	26.03	.95766	36.05	29.87	28.61	.95292	39.30	32.72	31.18
.96191	32.85	27.10	26.07	.95759	36.10	29.92	28.65	.95284	39.35	32.77	31.22
.96185	32.90	27.14	26.11	.95752	36.15	29.96	28.69	.95277	39.40	32.81	31.26
.96178	32.95	27.19	26.15	.95745	36.20	30.00	28.73	.95269	39.45	32.86	31.30
.96172	33.00	27.23	26.19	.95738	36.25	30.05	28.77	.95262	39.50	32.90	31.34
.96166	33.05	27.27	26.23	.95731	36.30	30.09	28.81	.95254	39.55	32.95	31.38
.96159	33.10	27.32	26.27	.95724	36.35	30.13	28.84	.95246	39.60	32.99	31.42
.96153	33.15	27.36	26.31	.95717	36.40	30.17	28.88	.95239	39.65	33.04	31.46
.96146	33.20	27.40	26.35	.95710	36.45	30.22	28.92	.95231	39.70	33.08	31.50
.96140	33.25	27.45	26.39	.95703	36.50	30.26	28.96	.95223	39.75	33.13	31.54
.96133	33.30	27.49	26.43	.95695	36.55	30.30	29.00	.95216	39.80	33.17	31.58
.96127	33.35	27.53	26.47	.95688	36.60	30.35	29.04	.95208	39.85	33.22	31.62
.96120	33.40	27.57	26.51	.95681	36.65	30.39	29.08	.95200	39.90	33.27	31.66
.96114	33.45	27.62	26.55	.95674	36.70	30.44	29.12	.95193	39.95	33.31	31.70
.96108	33.50	27.66	26.59	.95667	36.75	30.48	29.16	.95185	40.00	33.35	31.74
.96101	33.55	27.70	26.63	.95660	36.80	30.52	29.20	.95177	40.05	33.39	31.78
.96095	33.60	27.75	26.67	.95653	36.85	30.57	29.24	.95169	40.10	33.44	31.82
.96088	33.65	27.79	26.71	.95646	36.90	30.61	29.29	.95161	40.15	33.48	31.86
.96082	33.70	27.83	26.75	.95639	36.95	30.66	29.32	.95154	40.20	33.53	31.90
.96075	33.75	27.88	26.79	.95632	37.00	30.70	29.36	.95146	40.25	33.57	31.94
.96069	33.80	27.92	26.82	.95625	37.05	30.74	29.40	.95138	40.30	33.61	31.98
.96062	33.85	27.96	26.86	.95618	37.10	30.79	29.44	.95130	40.35	33.66	32.02
.96056	33.90	28.00	26.90	.95610	37.15	30.83	29.48	.95122	40.40	33.70	32.06
.96049	33.95	28.05	26.94	.95603	37.20	30.88	29.52	.95114	40.45	33.75	32.10
.96043	34.00	28.09	26.98	.95596	37.25	30.92	29.56	.95107	40.50	33.79	32.14
.96036	34.05	28.13	27.02	.95589	37.30	30.96	29.60	.95099	40.55	33.84	32.18
.96030	34.10	28.18	27.06	.95581	37.35	31.01	29.64	.95091	40.60	33.88	32.22
.96023	34.15	28.22	27.10	.95574	37.40	31.05	29.68	.95083	40.65	33.93	32.26
.96016	34.20	28.26	27.14	.95567	37.45	31.10	29.72	.95075	40.70	33.97	32.30
.96010	34.25	28.31	27.18	.95560	37.50	31.14	29.76	.95067	40.75	34.02	32.34
.96003	34.30	28.35	27.22	.95552	37.55	31.18	29.80	.95059	40.80	34.06	32.38
.95996	34.35	28.39	27.26	.95545	37.60	31.23	29.84	.95052	40.85	34.11	32.42
.95990	34.40	28.43	27.30	.95538	37.65	31.27	29.88	.95044	40.90	34.15	32.46
.95983	34.45	28.48	27.34	.95531	37.70	31.32	29.92	.95036	40.95	34.20	32.50
.95977	34.50	28.52	27.38	.95523	37.75	31.36	29.96	.95028	41.00	34.24	32.54
.95970	34.55	28.56	27.42	.95516	37.80	31.40	30.00	.95020	41.05	34.28	32.58
.95963	34.60	28.61	27.46	.95509	37.85	31.45	30.04	.95012	41.10	34.33	32.62
.95957	34.65	28.65	27.50	.95502	37.90	31.49	30.08	.95004	41.15	34.37	32.66
.95950	34.70	28.70	27.54	.95494	37.95	31.54	30.12	.94996	41.20	34.42	32.70

TABLE II.—*Percentage of alcohol*—Continued.

Specific gravity at 59° F.	Alcohol.			Specific gravity at 59° F.	Alcohol.			Specific gravity at 59° F.	Alcohol.		
	Per cent by vol-ume.	Per cent by weight.	Grams per 100 cc.		Per cent by vol-ume.	Per cent by weight.	Grams per 100 cc.		Per cent by vol-ume.	Per cent by weight.	Grams per 100 cc.
0.94988	41.25	34.46	32.74	0.94493	44.25	37.16	35.11	0.93962	47.25	39.90	37.49
.94980	41.30	34.50	32.78	.94484	44.30	37.21	35.15	.93953	47.30	39.95	37.53
.94972	41.35	34.55	32.82	.94476	44.35	37.25	35.19	.93944	47.35	39.99	37.57
.94964	41.40	34.59	32.86	.94467	44.40	37.30	35.23	.93934	47.40	40.04	37.61
.94956	41.45	34.64	32.90	.94459	44.45	37.34	35.27	.93925	47.45	40.08	37.65
.94948	41.50	34.68	32.93	.94450	44.50	37.39	35.31	.93916	47.50	40.13	37.69
.94940	41.55	34.73	32.97	.94441	44.55	37.44	35.35	.93906	47.55	40.18	37.73
.94932	41.60	34.77	33.01	.94433	44.60	37.48	35.39	.93898	47.60	40.22	37.77
.94924	41.65	34.82	33.05	.94424	44.65	37.53	35.43	.93888	47.65	40.27	37.81
.94916	41.70	34.86	33.09	.94416	44.70	37.57	35.47	.93879	47.70	40.32	37.85
.94908	41.75	34.91	33.13	.94407	44.75	37.62	35.51	.93870	47.75	40.37	37.89
.94900	41.80	34.95	33.17	.94398	44.80	37.66	35.55	.93861	47.80	40.41	37.93
.94892	41.85	35.00	33.21	.94390	44.85	37.71	35.59	.93852	47.85	40.46	37.97
.94884	41.90	35.04	33.25	.94381	44.90	37.76	35.63	.93842	47.90	40.51	38.01
.94876	41.95	35.09	33.29	.94373	44.95	37.80	35.67	.93833	47.95	40.55	38.05
.94868	42.00	35.13	33.33	.94364	45.00	37.84	35.71	.93824	48.00	40.60	38.09
.94860	42.05	35.18	33.37	.94355	45.05	37.89	35.75	.93815	48.05	40.65	38.13
.94852	42.10	35.22	33.41	.94346	45.10	37.93	35.79	.93806	48.10	40.69	38.17
.94843	42.15	35.27	33.45	.94338	45.15	37.98	35.83	.93796	48.15	40.74	38.21
.94835	42.20	35.31	33.49	.94329	45.20	38.02	35.87	.93786	48.20	40.78	38.25
.94827	42.25	35.36	33.53	.94320	45.25	38.07	35.91	.93777	48.25	40.83	38.29
.94810	42.30	35.40	33.57	.94311	45.30	38.12	35.95	.93768	48.30	40.88	38.33
.94811	42.35	35.45	33.61	.94302	45.35	38.16	35.99	.93758	48.35	40.92	38.37
.94802	42.40	35.49	33.65	.94294	45.40	38.21	36.03	.93749	48.40	40.97	38.41
.94794	42.45	35.54	33.69	.94285	45.45	38.25	36.07	.93739	48.45	41.01	38.45
.94786	42.50	35.58	33.73	.94276	45.50	38.30	36.11	.93730	48.50	41.06	38.49
.94778	42.55	35.63	33.77	.94267	45.55	38.35	36.15	.93721	48.55	41.11	38.53
.94770	42.60	35.67	33.81	.94258	45.60	38.39	36.19	.93711	48.60	41.15	38.57
.94761	42.65	35.72	33.85	.94250	45.65	38.44	36.23	.93702	48.65	41.20	38.61
.94753	42.70	35.76	33.89	.94241	45.70	38.48	36.26	.93692	48.70	41.24	38.65
.94745	42.75	35.81	33.93	.94232	45.75	38.53	36.30	.93683	48.75	41.29	38.69
.94737	42.80	35.85	33.97	.94223	45.80	38.57	36.34	.93674	48.80	41.34	38.72
.94729	42.85	35.90	34.00	.94214	45.85	38.62	36.38	.93664	48.85	41.38	38.76
.94720	42.90	35.94	34.04	.94206	45.90	38.66	36.42	.93655	48.90	41.43	38.80
.94712	42.95	35.99	34.08	.94197	45.95	38.71	36.46	.93645	48.95	41.47	38.84
.94704	43.00	36.03	34.12	.94188	46.00	38.75	36.50	.93636	49.00	41.52	38.88
.94696	43.05	36.08	34.16	.94179	46.05	38.80	36.54	.93626	49.05	41.57	38.92
.94687	43.10	36.12	34.20	.94170	46.10	38.84	36.58	.93617	49.10	41.61	38.96
.94679	43.15	36.17	34.24	.94161	46.15	38.89	36.62	.93607	49.15	41.66	39.00
.94670	43.20	36.21	34.28	.94152	46.20	38.93	36.66	.93598	49.20	41.71	39.04
.94662	43.25	36.25	34.32	.94143	46.25	38.98	36.70	.93588	49.25	41.76	39.08
.94654	43.30	36.30	34.36	.94134	46.30	39.03	36.74	.93578	49.30	41.80	39.12
.94645	43.35	36.35	34.40	.94125	46.35	39.07	36.78	.93569	49.35	41.85	39.16
.94637	43.40	36.39	34.44	.94116	46.40	39.12	36.82	.93559	49.40	41.90	39.20
.94628	43.45	36.44	34.48	.94107	46.45	39.16	36.86	.93550	49.45	41.94	39.24
.94620	43.50	36.48	34.52	.94098	46.50	39.21	36.90	.93540	49.50	41.99	39.28
.94612	43.55	36.53	34.56	.94089	46.55	39.26	36.94	.93530	49.55	42.04	39.32
.94603	43.60	36.57	34.60	.94080	46.60	39.30	36.98	.93521	49.60	42.08	39.36
.94595	43.65	36.62	34.64	.94071	46.65	39.35	37.02	.93511	49.65	42.13	39.40
.94586	43.70	36.66	34.68	.94062	46.70	39.39	37.06	.93502	49.70	42.18	39.44
.94578	43.75	36.71	34.72	.94053	46.75	39.44	37.09	.93492	49.75	42.23	39.48
.94570	43.80	36.75	34.76	.94044	46.80	39.49	37.13	.93482	49.80	42.27	39.52
.94561	43.85	36.80	34.80	.94035	46.85	39.53	37.17	.93473	49.85	42.32	39.56
.94553	43.90	36.84	34.84	.94026	46.90	39.58	37.21	.93463	49.90	42.37	39.60
.94544	43.95	36.89	34.88	.94017	46.95	39.62	37.25	.93454	49.95	42.41	39.63
.94536	44.00	36.93	34.91	.94008	47.00	39.67	37.29				
.94527	44.05	36.98	34.95	.93999	47.05	39.72	37.33				
.94519	44.10	37.02	34.99	.93990	47.10	39.76	37.37				
.94510	44.15	37.07	35.03	.93980	47.15	39.81	37.41				
.94502	44.20	37.11	35.07	.93971	47.20	39.85	37.45				

TABLE III.—*Extract in beer wort.*<sup>a</sup>

[According to Schultz and Ostermann.]

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0000	0.00	0.00	1.0065	1.69	1.70	1.0130	3.35	3.39	1.0195	5.06	5.16
1.0001	0.03	0.03	1.0066	1.72	1.73	1.0131	3.38	3.42	1.0196	5.09	5.19
1.0002	0.05	0.05	1.0067	1.74	1.75	1.0132	3.41	3.46	1.0197	5.12	5.22
1.0003	0.08	0.08	1.0068	1.77	1.78	1.0133	3.43	3.48	1.0198	5.15	5.25
1.0004	0.10	1.10	1.0069	1.79	1.80	1.0134	3.46	3.51	1.0199	5.17	5.27
1.0005	0.13	0.13	1.0070	1.82	1.83	1.0135	3.48	3.53	1.0200	5.20	5.30
1.0006	0.16	0.16	1.0071	1.84	1.85	1.0136	3.51	3.56	1.0201	5.23	5.34
1.0007	0.18	0.18	1.0072	1.87	1.88	1.0127	3.54	3.59	1.0202	5.25	5.36
1.0008	0.21	0.21	1.0073	1.90	1.91	1.0138	3.56	3.61	1.0203	5.28	5.39
1.0009	0.24	0.24	1.0074	1.92	1.93	1.0139	3.59	3.64	1.0204	5.30	5.41
1.0010	0.26	0.26	1.0075	1.95	1.96	1.0140	3.61	3.66	1.0205	5.33	5.44
1.0011	0.29	0.29	1.0076	1.97	1.98	1.0141	3.64	3.69	1.0206	5.35	5.46
1.0012	0.31	0.31	1.0077	2.00	2.02	1.0142	3.66	3.71	1.0207	5.38	5.49
1.0013	0.34	0.34	1.0078	2.02	2.04	1.0143	3.69	3.74	1.0208	5.40	5.51
1.0014	0.37	0.37	1.0079	2.05	2.07	1.0144	3.72	4.77	1.0209	5.43	5.54
1.0015	0.39	0.39	1.0080	2.07	2.09	1.0145	3.74	3.79	1.0210	5.45	5.56
1.0016	0.42	0.42	1.0081	2.10	2.12	1.0146	3.77	3.83	1.0211	5.48	5.60
1.0017	0.45	0.45	1.0082	2.12	2.14	1.0147	3.79	3.85	1.0212	5.50	5.62
1.0018	0.47	0.47	1.0083	2.15	2.17	1.0148	3.82	3.88	1.0213	5.53	5.65
1.0019	0.50	0.50	1.0084	2.17	2.19	1.0149	3.85	3.91	1.0214	5.55	5.67
1.0020	0.52	0.52	1.0085	2.20	2.22	1.0150	3.87	3.93	1.0215	5.57	5.69
1.0021	0.55	0.55	1.0086	2.23	2.25	1.0151	3.90	3.96	1.0216	5.60	5.72
1.0022	0.58	0.58	1.0087	2.25	2.27	1.0152	3.92	3.98	1.0217	5.62	5.74
1.0023	0.60	0.60	1.0088	2.28	2.30	1.0153	3.95	4.01	1.0218	5.65	5.77
1.0024	0.63	0.63	1.0089	2.30	2.32	1.0154	3.97	4.03	1.0219	5.67	5.79
1.0025	0.66	0.66	1.0090	2.33	2.35	1.0155	4.00	4.06	1.0220	5.70	5.83
1.0026	0.68	0.68	1.0091	2.35	2.37	1.0156	4.03	4.09	1.0221	5.72	5.85
1.0027	0.71	0.71	1.0092	2.38	2.40	1.0157	4.05	4.11	1.0222	5.75	5.88
1.0028	0.73	0.73	1.0093	2.41	2.43	1.0158	4.08	4.14	1.0223	5.77	5.90
1.0029	0.76	0.76	1.0094	2.43	2.45	1.0159	4.10	4.17	1.0224	5.80	5.93
1.0030	0.79	0.79	1.0095	2.46	2.48	1.0160	4.13	4.20	1.0225	5.82	5.95
1.0031	0.81	0.81	1.0096	2.48	2.50	1.0161	4.16	4.23	1.0226	5.84	5.97
1.0032	0.84	0.84	1.0097	2.51	2.53	1.0162	4.18	4.25	1.0227	5.87	6.00
1.0033	0.87	0.87	1.0098	2.53	2.55	1.0163	4.21	4.28	1.0228	5.89	6.02
1.0034	0.89	0.89	1.0099	2.56	2.59	1.0164	4.23	4.30	1.0229	5.92	6.06
1.0035	0.92	0.92	1.0100	2.58	2.61	1.0165	4.26	4.33	1.0230	5.94	6.08
1.0036	0.94	0.94	1.0101	2.61	2.64	1.0166	4.28	4.35	1.0231	5.97	6.11
1.0037	0.97	0.97	1.0102	2.64	2.67	1.0167	4.31	4.38	1.0232	5.99	6.13
1.0038	1.00	1.00	1.0103	2.66	2.69	1.0168	4.34	4.41	1.0233	6.02	6.16
1.0039	1.02	1.02	1.0104	2.69	2.72	1.0169	4.36	4.43	1.0234	6.04	6.18
1.0040	1.05	1.05	1.0105	2.71	2.74	1.0170	4.39	4.46	1.0235	6.07	6.21
1.0041	1.08	1.08	1.0106	2.74	2.77	1.0171	4.42	4.50	1.0236	6.09	6.23
1.0042	1.10	1.10	1.0107	2.76	2.79	1.0172	4.44	4.52	1.0237	6.11	6.25
1.0043	1.13	1.13	1.0108	2.79	2.82	1.0173	4.47	4.55	1.0238	6.14	6.29
1.0044	1.15	1.16	1.0109	2.82	2.85	1.0174	4.50	4.58	1.0239	6.16	6.31
1.0045	1.18	1.19	1.0110	2.84	2.87	1.0175	4.53	4.61	1.0240	6.19	6.34
1.0046	1.21	1.22	1.0111	2.87	2.90	1.0176	4.55	4.63	1.0241	6.21	6.36
1.0047	1.23	1.24	1.0112	2.89	2.92	1.0177	4.58	4.66	1.0242	6.24	6.39
1.0048	1.26	1.27	1.0113	2.92	2.95	1.0178	4.61	4.69	1.0243	6.26	6.41
1.0049	1.29	1.30	1.0114	2.94	2.97	1.0179	4.63	4.71	1.0244	6.29	6.44
1.0050	1.31	1.32	1.0115	2.97	3.00	1.0180	4.66	4.74	1.0245	6.31	6.46
1.0051	1.34	1.35	1.0116	2.99	3.02	1.0181	4.69	4.77	1.0246	6.34	6.50
1.0052	1.36	1.37	1.0117	3.02	3.06	1.0182	4.71	4.80	1.0247	6.36	6.52
1.0053	1.39	1.40	1.0118	3.05	3.09	1.0183	4.74	4.83	1.0248	6.39	6.55
1.0054	1.41	1.42	1.0119	3.07	3.11	1.0184	4.77	4.86	1.0249	6.41	6.57
1.0055	1.44	1.45	1.0120	3.10	3.14	1.0185	4.79	4.88	1.0250	6.44	6.60
1.0056	1.46	1.47	1.0121	3.12	3.16	1.0186	4.82	4.91	1.0251	6.47	6.63
1.0057	1.49	1.50	1.0122	3.15	3.19	1.0187	4.85	4.94	1.0252	6.50	6.66
1.0058	1.51	1.52	1.0123	3.17	3.21	1.0188	4.88	4.97	1.0253	6.52	6.68
1.0059	1.54	1.55	1.0124	3.20	3.24	1.0189	4.90	4.99	1.0254	6.55	6.72
1.0060	1.56	1.57	1.0125	3.23	3.27	1.0190	4.93	5.02	1.0255	6.58	6.75
1.0061	1.59	1.60	1.0126	3.25	3.29	1.0191	4.96	5.05	1.0256	6.61	6.78
1.0062	1.62	1.63	1.0127	3.28	3.32	1.0192	4.98	5.08	1.0257	6.63	6.80
1.0063	1.64	1.65	1.0128	3.30	3.34	1.0193	5.01	5.11	1.0258	6.66	6.83
1.0064	1.67	1.68	1.0129	3.33	3.37	1.0194	5.04	5.14	1.0259	6.69	6.86

<sup>a</sup>Calculated from results obtained by drying below 75° C.

TABLE III.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0260	6.71	6.88	1.0325	8.27	8.54	1.0390	9.92	10.31	1.0455	11.53	12.05
1.0261	6.74	6.92	1.0326	8.29	8.56	1.0391	9.95	10.34	1.0456	11.55	12.08
1.0262	6.77	6.95	1.0327	8.32	8.59	1.0392	9.97	10.36	1.0457	11.57	12.10
1.0263	6.80	6.98	1.0328	8.34	8.61	1.0393	9.99	10.38	1.0458	11.60	12.13
1.0264	6.82	7.00	1.0329	8.37	8.65	1.0394	10.02	10.41	1.0459	11.62	12.15
1.0265	6.85	7.03	1.0330	8.40	8.68	1.0395	10.04	10.44	1.0460	11.65	12.19
1.0266	6.88	7.06	1.0331	8.43	8.71	1.0396	10.06	10.46	1.0461	11.67	12.21
1.0267	6.91	7.09	1.0332	8.45	8.73	1.0397	10.09	10.49	1.0462	11.70	12.24
1.0268	6.93	7.12	1.0333	8.48	8.76	1.0398	10.11	10.51	1.0463	11.72	12.26
1.0269	6.96	7.15	1.0334	8.51	8.79	1.0399	10.13	10.53	1.0464	11.75	12.30
1.0270	6.99	7.18	1.0335	8.53	8.82	1.0400	10.16	10.57	1.0465	11.77	12.32
1.0271	7.01	7.20	1.0336	8.56	8.85	1.0401	10.18	10.59	1.0466	11.79	12.34
1.0272	7.04	7.23	1.0337	8.59	8.88	1.0402	10.20	10.61	1.0467	11.82	12.37
1.0273	7.07	7.26	1.0338	8.61	8.90	1.0403	10.23	10.64	1.0468	11.84	12.39
1.0274	7.10	7.29	1.0339	8.64	8.93	1.0404	10.25	10.66	1.0469	11.87	12.43
1.0275	7.12	7.32	1.0340	8.67	8.96	1.0405	10.27	10.69	1.0470	11.89	12.45
1.0276	7.15	7.35	1.0341	8.70	9.00	1.0406	10.30	10.72	1.0471	11.92	12.48
1.0277	7.18	7.38	1.0342	8.72	9.02	1.0407	10.32	10.74	1.0472	11.94	12.50
1.0278	7.21	7.41	1.0343	8.75	9.05	1.0408	10.35	10.77	1.0473	11.97	12.54
1.0279	7.23	7.43	1.0344	8.78	9.08	1.0409	10.37	10.79	1.0474	11.99	12.56
1.0280	7.26	7.46	1.0345	8.80	9.10	1.0410	10.40	10.83	1.0475	12.01	12.58
1.0281	7.28	7.48	1.0346	8.83	9.14	1.0411	10.42	10.85	1.0476	12.04	12.61
1.0282	7.30	7.51	1.0347	8.86	9.17	1.0412	10.45	10.88	1.0477	12.06	12.64
1.0283	7.33	7.54	1.0348	8.88	9.19	1.0413	10.47	10.90	1.0478	12.09	12.67
1.0284	7.35	7.56	1.0349	8.91	9.22	1.0414	10.50	10.93	1.0479	12.11	12.69
1.0285	7.37	7.58	1.0350	8.94	9.25	1.0415	10.52	10.96	1.0480	12.14	12.72
1.0286	7.39	7.60	1.0351	8.97	9.28	1.0416	10.55	10.99	1.0481	12.16	12.74
1.0287	7.42	7.63	1.0352	8.99	9.31	1.0417	10.57	11.01	1.0482	12.19	12.78
1.0288	7.44	7.65	1.0353	9.02	9.34	1.0418	10.60	11.04	1.0483	12.21	12.80
1.0289	7.46	7.68	1.0354	9.05	9.37	1.0419	10.62	11.06	1.0484	12.23	12.82
1.0290	7.48	7.70	1.0355	9.07	9.39	1.0420	10.65	11.10	1.0485	12.26	12.85
1.0291	7.51	7.73	1.0356	9.10	9.42	1.0421	10.67	11.12	1.0486	12.28	12.88
1.0292	7.53	7.75	1.0357	9.13	9.46	1.0422	10.70	11.15	1.0487	12.31	12.91
1.0293	7.55	7.77	1.0358	9.15	9.48	1.0423	10.72	11.17	1.0488	12.33	12.93
1.0294	7.57	7.79	1.0359	9.18	9.51	1.0424	10.75	11.21	1.0489	12.36	12.96
1.0295	7.60	7.82	1.0360	9.21	9.54	1.0425	10.77	11.23	1.0490	12.38	12.99
1.0296	7.62	7.85	1.0361	9.24	9.57	1.0426	10.80	11.26	1.0491	12.41	13.02
1.0297	7.64	7.87	1.0362	9.26	9.60	1.0427	10.82	11.28	1.0492	12.43	13.04
1.0298	7.66	7.89	1.0363	9.29	9.63	1.0428	10.85	11.31	1.0493	12.45	13.06
1.0299	7.69	7.92	1.0364	9.31	9.65	1.0429	10.88	11.35	1.0494	12.48	13.10
1.0300	7.71	7.94	1.0365	9.34	9.68	1.0430	10.90	11.37	1.0495	12.50	13.12
1.0301	7.73	7.96	1.0366	9.36	9.70	1.0431	10.93	11.40	1.0496	12.53	13.15
1.0302	7.75	7.98	1.0367	9.38	9.72	1.0432	10.95	11.42	1.0497	12.55	13.17
1.0303	7.77	8.01	1.0368	9.41	9.76	1.0433	10.98	11.46	1.0498	12.58	13.21
1.0304	7.80	8.04	1.0369	9.43	9.78	1.0434	11.00	11.48	1.0499	12.60	13.23
1.0305	7.82	8.06	1.0370	9.45	9.80	1.0435	11.03	11.51	1.0500	12.63	13.26
1.0306	7.84	8.08	1.0371	9.48	9.83	1.0436	11.05	11.53	1.0501	12.65	13.28
1.0307	7.86	8.10	1.0372	9.50	9.85	1.0437	11.08	11.56	1.0502	12.67	13.31
1.0308	7.89	8.13	1.0373	9.52	9.88	1.0438	11.10	11.59	1.0503	12.70	13.34
1.0309	7.91	8.15	1.0374	9.55	9.91	1.0439	11.13	11.62	1.0504	12.72	13.36
1.0310	7.93	8.18	1.0375	9.57	9.93	1.0440	11.15	11.64	1.0505	12.75	13.39
1.0311	7.95	8.20	1.0376	9.59	9.95	1.0441	11.18	11.67	1.0506	12.77	13.42
1.0312	7.98	8.23	1.0377	9.62	9.98	1.0442	11.20	11.70	1.0507	12.80	13.45
1.0313	8.00	8.25	1.0378	9.64	10.00	1.0443	11.23	11.73	1.0508	12.82	13.47
1.0314	8.02	8.27	1.0379	9.66	10.03	1.0444	11.25	11.75	1.0509	12.85	13.50
1.0315	8.04	8.29	1.0380	9.69	10.06	1.0445	11.28	11.78	1.0510	12.87	13.53
1.0316	8.07	8.33	1.0381	9.71	10.08	1.0446	11.30	11.80	1.0511	12.90	13.56
1.0317	8.09	8.35	1.0382	9.73	10.10	1.0447	11.33	11.84	1.0512	12.92	13.58
1.0318	8.11	8.37	1.0383	9.76	10.13	1.0448	11.35	11.86	1.0513	12.94	13.60
1.0319	8.13	8.39	1.0384	9.78	10.16	1.0449	11.38	11.89	1.0514	12.97	13.64
1.0320	8.16	8.42	1.0385	9.81	10.19	1.0450	11.40	11.91	1.0515	12.99	13.66
1.0321	8.18	8.44	1.0386	9.83	10.21	1.0451	11.43	11.95	1.0516	13.02	13.69
1.0322	8.20	8.46	1.0387	9.85	10.23	1.0452	11.45	11.97	1.0517	13.04	13.71
1.0323	8.22	8.49	1.0388	9.88	10.26	1.0453	11.48	12.00	1.0518	13.07	13.75
1.0324	8.25	8.52	1.0389	9.90	10.29	1.0454	11.50	12.02	1.0519	13.09	13.77



TABLE III.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0520	13.12	13.80	1.0585	14.75	15.61	1.0650	16.25	17.31	1.0715	17.81	19.08
1.0521	13.14	13.82	1.0586	14.78	15.65	1.0651	16.27	17.33	1.0716	17.84	19.12
1.0522	13.16	13.85	1.0587	14.81	15.68	1.0652	16.30	17.36	1.0717	17.86	19.14
1.0523	13.19	13.88	1.0588	14.83	15.70	1.0653	16.32	17.39	1.0718	17.88	19.16
1.0524	13.21	13.90	1.0589	14.86	15.74	1.0654	16.35	17.42	1.0719	17.90	19.19
1.0525	13.24	13.94	1.0590	14.89	15.77	1.0655	16.37	17.44	1.0720	17.93	19.22
1.0526	13.26	13.96	1.0591	14.91	15.79	1.0656	16.40	17.48	1.0721	17.95	19.24
1.0527	13.29	13.99	1.0592	14.94	15.82	1.0657	16.42	17.50	1.0722	17.97	19.27
1.0528	13.31	14.01	1.0593	14.96	15.85	1.0658	16.45	17.53	1.0723	17.99	19.29
1.0529	13.34	14.05	1.0594	14.99	15.88	1.0659	16.47	17.56	1.0724	18.02	19.32
1.0530	13.36	14.07	1.0595	15.02	15.91	1.0660	16.50	17.59	1.0725	18.04	19.35
1.0531	13.38	14.09	1.0596	15.04	15.94	1.0661	16.52	17.61	1.0726	18.06	19.37
1.0532	13.41	14.12	1.0597	15.07	15.97	1.0662	16.54	17.63	1.0727	18.08	19.39
1.0533	13.43	14.15	1.0598	15.09	15.99	1.0663	16.57	17.67	1.0728	18.11	19.43
1.0534	13.46	14.18	1.0599	15.11	16.02	1.0664	16.59	17.69	1.0729	18.13	19.45
1.0535	13.48	14.20	1.0600	15.14	16.05	1.0665	16.62	17.73	1.0730	18.15	19.47
1.0536	13.51	14.23	1.0601	15.16	16.07	1.0666	16.64	17.75	1.0731	18.17	19.50
1.0537	13.53	14.26	1.0602	15.18	16.09	1.0667	16.67	17.78	1.0732	18.20	19.53
1.0538	13.56	14.29	1.0603	15.20	16.12	1.0668	16.69	17.80	1.0733	18.22	19.55
1.0539	13.58	14.31	1.0604	15.23	16.15	1.0669	16.72	17.84	1.0734	18.24	19.58
1.0540	13.61	14.34	1.0605	15.25	16.17	1.0670	16.74	17.86	1.0735	18.26	19.60
1.0541	13.63	14.37	1.0606	15.27	16.20	1.0671	16.76	17.88	1.0736	18.29	19.64
1.0542	13.66	14.40	1.0607	15.29	16.22	1.0672	16.79	17.92	1.0737	18.31	19.66
1.0543	13.68	14.42	1.0608	15.31	16.24	1.0673	16.81	17.94	1.0738	18.33	19.68
1.0544	13.71	14.46	1.0609	15.34	16.27	1.0674	16.84	17.98	1.0739	18.35	19.71
1.0545	13.73	14.48	1.0610	15.36	16.30	1.0675	16.86	18.00	1.0740	18.38	19.74
1.0546	13.76	14.51	1.0611	15.38	16.32	1.0676	16.89	18.03	1.0741	18.40	19.76
1.0547	13.78	14.53	1.0612	15.40	16.34	1.0677	16.91	18.05	1.0742	18.42	19.79
1.0548	13.81	14.57	1.0613	15.43	16.38	1.0678	16.94	18.09	1.0743	18.44	19.81
1.0549	13.83	14.59	1.0614	15.45	16.40	1.0679	16.96	18.11	1.0744	18.47	19.84
1.0550	13.86	14.62	1.0615	15.47	16.42	1.0680	16.99	18.15	1.0745	18.49	19.87
1.0551	13.88	14.64	1.0616	15.49	16.44	1.0681	17.01	18.17	1.0746	18.51	19.89
1.0552	13.91	14.68	1.0617	15.52	16.48	1.0682	17.03	18.19	1.0747	18.53	19.91
1.0553	13.93	14.70	1.0618	15.54	16.50	1.0683	17.06	18.23	1.0748	18.55	19.94
1.0554	13.96	14.73	1.0619	15.56	16.52	1.0684	17.08	18.25	1.0749	18.57	19.96
1.0555	13.98	14.76	1.0620	15.58	16.55	1.0685	17.11	18.28	1.0750	18.59	19.98
1.0556	14.01	14.79	1.0621	15.60	16.57	1.0686	17.13	18.31	1.0751	18.62	20.02
1.0557	14.03	14.81	1.0622	15.63	16.60	1.0687	17.16	18.34	1.0752	18.64	20.04
1.0558	14.06	14.84	1.0623	15.65	16.62	1.0688	17.18	18.36	1.0753	18.66	20.07
1.0559	14.08	14.87	1.0624	15.67	16.64	1.0689	17.21	18.40	1.0754	18.68	20.09
1.0560	14.11	14.90	1.0625	15.69	16.66	1.0690	17.23	18.42	1.0755	18.70	20.11
1.0561	14.13	14.92	1.0626	15.72	16.70	1.0691	17.25	18.44	1.0756	18.72	20.14
1.0562	14.16	14.96	1.0627	15.74	16.73	1.0692	17.28	18.48	1.0757	18.74	20.16
1.0563	14.18	14.98	1.0628	15.76	16.75	1.0693	17.30	18.50	1.0758	18.76	20.18
1.0564	14.21	15.01	1.0629	15.78	16.77	1.0694	17.33	18.53	1.0759	18.78	20.21
1.0565	14.23	15.03	1.0630	15.80	16.80	1.0695	17.35	18.56	1.0760	18.81	20.24
1.0566	14.26	15.07	1.0631	15.83	16.83	1.0696	17.38	18.59	1.0761	18.83	20.26
1.0567	14.28	15.09	1.0632	15.85	16.85	1.0697	17.40	18.61	1.0762	18.85	20.29
1.0568	14.31	15.12	1.0633	15.87	16.87	1.0698	17.43	18.65	1.0763	18.87	20.31
1.0569	14.33	15.15	1.0634	15.89	16.90	1.0699	17.45	18.67	1.0764	18.89	20.33
1.0570	14.36	15.18	1.0635	15.92	16.93	1.0700	17.48	18.70	1.0765	18.91	20.36
1.0571	14.38	15.20	1.0636	15.94	16.95	1.0701	17.50	18.73	1.0766	18.93	20.38
1.0572	14.41	15.23	1.0637	15.96	16.98	1.0702	17.52	18.75	1.0767	18.95	20.40
1.0573	14.44	15.27	1.0638	15.98	17.00	1.0703	17.54	18.77	1.0768	18.97	20.43
1.0574	14.46	15.29	1.0639	16.01	17.03	1.0704	17.57	18.81	1.0769	19.00	20.46
1.0575	14.49	15.32	1.0640	16.03	17.06	1.0705	17.59	18.83	1.0770	19.02	20.48
1.0576	14.52	15.36	1.0641	16.05	17.08	1.0706	17.61	18.85	1.0771	19.04	20.51
1.0577	14.54	15.38	1.0642	16.07	17.10	1.0707	17.63	18.88	1.0772	19.06	20.53
1.0578	14.57	15.41	1.0643	16.09	17.12	1.0708	17.66	18.91	1.0773	19.08	20.55
1.0579	14.59	15.43	1.0644	16.12	17.16	1.0709	17.68	18.93	1.0774	19.10	20.58
1.0580	14.62	15.47	1.0645	16.14	17.18	1.0710	17.70	18.96	1.0775	19.12	20.60
1.0581	14.65	15.50	1.0646	16.16	17.20	1.0711	17.72	18.98	1.0776	19.14	20.63
1.0582	14.67	15.52	1.0647	16.18	17.23	1.0712	17.75	19.01	1.0777	19.17	20.66
1.0583	14.70	15.56	1.0648	16.21	17.26	1.0713	17.77	19.04	1.0778	19.19	20.68
1.0584	14.73	15.59	1.0649	16.23	17.28	1.0714	17.79	19.06	1.0779	19.21	20.71

TABLE III.—*Extract in beer wort*—Continued.

Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0780	19.23	20.73	1.0845	20.70	22.45	1.0910	22.19	24.21	1.0975	23.59	25.89
1.0781	19.25	20.75	1.0846	20.73	22.48	1.0911	22.21	24.24	1.0976	23.61	25.92
1.0782	19.27	20.78	1.0847	20.75	22.50	1.0912	22.23	24.26	1.0977	23.63	25.94
1.0783	19.29	20.80	1.0848	20.77	22.53	1.0913	22.26	24.29	1.0978	23.65	25.97
1.0784	19.31	20.82	1.0849	20.79	22.55	1.0914	22.28	24.31	1.0979	23.67	25.99
1.0785	19.33	20.85	1.0850	20.81	22.58	1.0915	22.30	24.34	1.0980	23.69	26.01
1.0786	19.36	20.88	1.0851	20.83	22.61	1.0916	22.32	24.37	1.0981	23.71	26.04
1.0787	19.38	20.90	1.0852	20.86	22.64	1.0917	22.34	24.39	1.0982	23.73	26.06
1.0788	19.40	20.93	1.0853	20.88	22.66	1.0918	22.37	24.42	1.0983	23.76	26.09
1.0789	19.42	20.95	1.0854	20.90	22.68	1.0919	22.39	24.44	1.0984	23.78	26.11
1.0790	19.44	20.98	1.0855	20.93	22.72	1.0920	22.41	24.47	1.0985	23.80	26.14
1.0791	19.46	21.00	1.0856	20.95	22.75	1.0921	22.43	24.49	1.0986	23.82	26.17
1.0792	19.49	21.03	1.0857	20.98	22.78	1.0922	22.45	24.51	1.0987	23.84	26.19
1.0793	19.51	21.06	1.0858	21.01	22.81	1.0923	22.48	24.54	1.0988	23.86	26.22
1.0794	19.53	21.08	1.0859	21.04	22.84	1.0924	22.50	24.56	1.0989	23.88	26.24
1.0795	19.56	21.11	1.0860	21.06	22.87	1.0925	22.52	24.60	1.0990	23.90	26.27
1.0796	19.58	21.14	1.0861	21.09	22.90	1.0926	22.54	24.62	1.0991	23.92	26.30
1.0797	19.60	21.16	1.0862	21.11	22.93	1.0927	22.56	24.64	1.0992	23.94	26.32
1.0798	19.63	21.20	1.0863	21.13	22.96	1.0928	22.59	24.67	1.0993	23.97	26.35
1.0799	19.65	21.22	1.0864	21.16	22.99	1.0929	22.61	24.70	1.0994	23.99	26.37
1.0800	19.67	21.24	1.0865	21.19	23.02	1.0930	22.63	24.73	1.0995	24.01	26.40
1.0801	19.70	21.28	1.0866	21.22	23.06	1.0931	22.65	24.76	1.0996	24.03	26.42
1.0802	19.72	21.30	1.0867	21.25	23.09	1.0932	22.67	24.78	1.0997	24.05	26.44
1.0803	19.74	21.33	1.0868	21.28	23.12	1.0933	22.69	24.81	1.0998	24.07	26.47
1.0804	19.77	21.36	1.0869	21.30	23.15	1.0934	22.71	24.83	1.0999	24.09	26.49
1.0805	19.79	21.38	1.0870	21.33	23.18	1.0935	22.73	24.86	1.1000	24.11	26.52
1.0806	19.81	21.41	1.0871	21.35	23.21	1.0936	22.75	24.89	1.1001	24.13	26.55
1.0807	19.84	21.43	1.0872	21.37	23.23	1.0937	22.77	24.91	1.1002	24.15	26.57
1.0808	19.86	21.46	1.0873	21.39	23.26	1.0938	22.80	24.93	1.1003	24.17	26.60
1.0809	19.88	21.49	1.0874	21.41	23.28	1.0939	22.82	24.96	1.1004	24.19	26.62
1.0810	19.91	21.52	1.0875	21.43	23.31	1.0940	22.84	24.99	1.1005	24.21	26.65
1.0811	19.93	21.55	1.0876	21.45	23.33	1.0941	22.86	25.01	1.1006	24.23	26.68
1.0812	19.96	21.58	1.0877	21.47	23.36	1.0942	22.88	25.03	1.1007	24.25	26.70
1.0813	19.98	21.60	1.0878	21.49	23.38	1.0943	22.90	25.06	1.1008	24.28	26.73
1.0814	20.00	21.63	1.0879	21.51	23.40	1.0944	22.92	25.08	1.1009	24.30	26.75
1.0815	20.03	21.66	1.0880	21.54	23.43	1.0945	22.94	25.11	1.1010	24.32	26.78
1.0816	20.05	21.69	1.0881	21.56	23.45	1.0946	22.96	25.14	1.1011	24.34	26.81
1.0817	20.07	21.71	1.0882	21.58	23.48	1.0947	22.98	25.16	1.1012	24.36	26.83
1.0818	20.10	21.74	1.0883	21.60	23.50	1.0948	23.00	25.18	1.1013	24.39	26.86
1.0819	20.12	21.77	1.0884	21.62	23.52	1.0949	23.03	25.21	1.1014	24.41	26.88
1.0820	20.14	21.79	1.0885	21.64	23.55	1.0950	23.05	25.24	1.1015	24.43	26.91
1.0821	20.17	21.83	1.0886	21.66	23.58	1.0951	23.07	25.26	1.1016	24.45	26.93
1.0822	20.19	21.85	1.0887	21.68	23.60	1.0952	23.10	25.29	1.1017	24.47	26.95
1.0823	20.21	21.87	1.0888	21.71	23.63	1.0953	23.12	25.31	1.1018	24.49	26.98
1.0824	20.24	21.91	1.0889	21.73	23.66	1.0954	23.14	25.34	1.1019	24.51	27.00
1.0825	20.26	21.93	1.0890	21.75	23.69	1.0955	23.16	25.37	1.1020	24.53	27.03
1.0826	20.28	21.96	1.0891	21.77	23.72	1.0956	23.18	25.39	1.1021	24.55	27.06
1.0827	20.31	21.99	1.0892	21.79	23.74	1.0957	23.20	25.42	1.1022	24.57	27.08
1.0828	20.33	22.01	1.0893	21.82	23.77	1.0958	23.23	25.45	1.1023	24.60	27.11
1.0829	20.35	22.04	1.0894	21.84	23.79	1.0959	23.25	25.47	1.1024	24.62	27.14
1.0830	20.37	22.06	1.0895	21.86	23.82	1.0960	23.27	25.50	1.1025	24.64	27.17
1.0831	20.39	22.08	1.0896	21.89	23.85	1.0961	23.29	25.53	1.1026	24.66	27.19
1.0832	20.41	22.11	1.0897	21.91	23.87	1.0962	23.31	25.55	1.1027	24.68	27.21
1.0833	20.43	22.13	1.0898	21.93	23.90	1.0963	23.33	25.58	1.1028	24.70	27.24
1.0834	20.46	22.16	1.0899	21.96	23.93	1.0964	23.35	25.60	1.1029	24.72	27.26
1.0835	20.48	22.19	1.0900	21.98	23.96	1.0965	23.37	25.63	1.1030	24.74	27.29
1.0836	20.50	22.21	1.0901	22.00	23.98	1.0966	23.39	25.66	1.1031	24.76	27.32
1.0837	20.52	22.24	1.0902	22.02	24.01	1.0967	23.41	25.68	1.1032	24.78	27.34
1.0838	20.54	22.26	1.0903	22.04	24.03	1.0968	23.44	25.71	1.1033	24.81	27.37
1.0839	20.56	22.29	1.0904	22.06	24.05	1.0969	23.46	25.73	1.1034	24.83	27.39
1.0840	20.59	22.32	1.0905	22.08	24.08	1.0970	23.48	25.76	1.1035	24.85	27.42
1.0841	20.62	22.35	1.0906	22.10	24.11	1.0971	23.50	25.79	1.1036	24.87	27.45
1.0842	20.64	22.38	1.0907	22.12	24.13	1.0972	23.52	25.81	1.1037	24.89	27.47
1.0843	20.66	22.40	1.0908	22.15	24.16	1.0973	23.55	25.84	1.1038	24.92	27.50
1.0844	20.68	22.42	1.0909	22.17	24.18	1.0974	23.57	25.86	1.1039	24.94	27.53

TABLE III.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.1040	24.96	27.56	1.1095	26.16	29.03	1.1150	27.29	30.43	1.1205	28.38	31.81
1.1041	24.98	27.58	1.1096	26.18	29.06	1.1151	27.31	30.45	1.1206	28.40	31.83
1.1042	25.00	27.60	1.1097	26.20	29.08	1.1152	27.33	30.47	1.1207	28.42	31.86
1.1043	25.03	27.63	1.1098	26.23	29.11	1.1153	27.35	30.50	1.1208	28.44	31.88
1.1044	25.05	27.66	1.1099	26.25	29.13	1.1154	27.37	30.52	1.1209	28.46	31.90
1.1045	25.07	27.69	1.1100	26.27	29.16	1.1155	27.38	30.55	1.1210	28.48	31.93
1.1046	25.09	27.72	1.1101	26.29	29.19	1.1156	27.40	30.57	1.1211	28.50	31.95
1.1047	25.11	27.74	1.1102	26.31	29.21	1.1157	27.42	30.59	1.1212	28.52	31.98
1.1048	25.14	27.77	1.1103	26.33	29.24	1.1158	27.44	30.62	1.1213	28.54	32.00
1.1049	25.16	27.79	1.1104	26.35	29.26	1.1159	27.46	30.64	1.1214	28.56	32.03
1.1050	25.18	27.82	1.1105	26.37	29.29	1.1160	27.48	30.67	1.1215	28.58	32.05
1.1051	25.20	27.85	1.1106	26.39	29.32	1.1161	27.50	30.69	1.1216	28.60	32.08
1.1052	25.22	27.87	1.1107	26.41	29.34	1.1162	27.52	30.72	1.1217	28.62	32.11
1.1053	25.24	27.90	1.1108	26.44	29.37	1.1163	27.54	30.75	1.1218	28.64	32.13
1.1054	25.27	27.93	1.1109	26.46	29.39	1.1164	27.56	30.77	1.1219	28.66	32.15
1.1055	25.29	27.96	1.1110	26.48	29.42	1.1165	27.58	30.80	1.1220	28.68	32.18
1.1056	25.31	27.98	1.1111	26.50	29.44	1.1166	27.60	30.82	1.1221	28.70	32.20
1.1057	25.33	28.00	1.1112	26.52	29.46	1.1167	27.62	30.85	1.1222	28.72	32.23
1.1058	25.35	28.03	1.1113	26.54	29.49	1.1168	27.64	30.87	1.1223	28.74	32.25
1.1059	25.38	28.06	1.1114	26.56	29.51	1.1169	27.66	30.89	1.1224	28.76	32.27
1.1060	25.40	28.09	1.1115	26.58	29.54	1.1170	27.68	30.92	1.1225	28.78	32.30
1.1061	25.42	28.12	1.1116	26.60	29.57	1.1171	27.70	30.94	1.1226	28.80	32.32
1.1062	25.44	28.14	1.1117	26.62	29.59	1.1172	27.72	30.97	1.1227	28.82	32.35
1.1063	25.46	28.17	1.1118	26.64	29.61	1.1173	27.74	31.00	1.1228	28.84	32.37
1.1064	25.48	28.19	1.1119	26.66	29.64	1.1174	27.76	31.02	1.1229	28.86	32.40
1.1065	25.50	28.22	1.1120	26.68	29.67	1.1175	27.78	31.05	1.1230	28.88	32.43
1.1066	25.52	28.25	1.1121	26.70	29.69	1.1176	27.80	31.07	1.1231	28.90	32.45
1.1067	25.54	28.27	1.1122	26.72	29.71	1.1177	27.82	31.09	1.1232	28.92	32.48
1.1068	25.57	28.30	1.1123	26.75	29.74	1.1178	27.84	31.12	1.1233	28.94	32.50
1.1069	25.59	28.32	1.1124	26.77	29.77	1.1179	27.86	31.15	1.1234	28.96	32.53
1.1070	25.61	28.35	1.1125	26.79	29.80	1.1180	27.88	31.18	1.1235	28.98	32.56
1.1071	25.63	28.38	1.1126	26.81	29.83	1.1181	27.90	31.20	1.1236	29.00	32.58
1.1072	25.65	28.40	1.1127	26.83	29.85	1.1182	27.92	31.23	1.1237	29.02	32.60
1.1073	25.67	28.43	1.1128	26.85	29.88	1.1183	27.94	31.25	1.1238	29.04	32.63
1.1074	25.69	28.45	1.1129	26.87	29.90	1.1184	27.96	31.27	1.1239	29.06	32.65
1.1075	25.71	28.48	1.1130	26.89	29.93	1.1185	27.98	31.30	1.1240	29.08	32.68
1.1076	25.73	28.51	1.1131	26.91	29.95	1.1186	28.00	31.32	1.1241	29.10	32.71
1.1077	25.75	28.53	1.1132	26.93	29.97	1.1187	28.02	31.35	1.1242	29.12	32.73
1.1078	25.78	28.56	1.1133	26.95	30.00	1.1188	28.04	31.37	1.1243	29.14	32.76
1.1079	25.80	28.58	1.1134	26.97	30.02	1.1189	28.07	31.40	1.1244	29.16	32.78
1.1080	25.82	28.61	1.1135	26.99	30.06	1.1190	28.09	31.43	1.1245	29.18	32.81
1.1081	25.84	28.64	1.1136	27.01	30.08	1.1191	28.11	31.45	1.1246	29.20	32.83
1.1082	25.86	28.66	1.1137	27.03	30.10	1.1192	28.13	31.48	1.1247	29.22	32.86
1.1083	25.89	28.69	1.1138	27.05	30.13	1.1193	28.15	31.51	1.1248	29.24	32.89
1.1084	25.91	28.72	1.1139	27.07	30.15	1.1194	28.17	31.53	1.1249	29.26	32.91
1.1085	25.93	28.75	1.1140	27.09	30.18	1.1195	28.19	31.56	1.1250	29.28	32.94
1.1086	25.96	28.78	1.1141	27.11	30.20	1.1196	28.21	31.59	1.1251	29.30	32.96
1.1087	25.98	28.80	1.1142	27.13	30.22	1.1197	28.23	31.61	1.1252	29.32	32.99
1.1088	26.01	28.83	1.1143	27.15	30.25	1.1198	28.25	31.63	1.1253	29.34	33.02
1.1089	26.03	28.86	1.1144	27.17	30.27	1.1199	28.27	31.65	1.1254	29.36	33.04
1.1090	26.05	28.89	1.1145	27.19	30.31	1.1200	28.28	31.68	1.1255	29.38	33.07
1.1091	26.07	28.92	1.1146	27.21	30.33	1.1201	28.30	31.70	1.1256	29.40	33.09
1.1092	26.09	28.94	1.1147	27.23	30.35	1.1202	28.32	31.73	1.1257	29.42	33.12
1.1093	26.12	28.97	1.1148	27.25	30.37	1.1203	28.34	31.75	1.1258	29.45	33.15
1.1094	26.14	29.00	1.1149	27.27	30.40	1.1204	28.36	31.78	1.1259	29.47	33.17

TABLE IV.—*Extract in beer wort.* <sup>a</sup>

[According to H. Ellion.]

Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0001	0.02	0.02	1.0066	1.63	1.64	1.0131	3.22	3.26	1.0196	4.79	4.89
1.0002	0.05	0.05	1.0067	1.66	1.67	1.0132	3.25	3.29	1.0197	4.82	4.91
1.0003	0.07	0.07	1.0068	1.68	1.69	1.0133	3.27	3.31	1.0198	4.84	4.94
1.0004	0.10	0.10	1.0069	1.71	1.72	1.0134	3.29	3.34	1.0199	4.87	4.96
1.0005	0.12	0.12	1.0070	1.73	1.74	1.0135	3.32	3.36	1.0200	4.89	4.99
1.0006	0.15	0.15	1.0071	1.76	1.77	1.0136	3.34	3.39	1.0201	4.91	5.01
1.0007	0.17	0.17	1.0072	1.78	1.79	1.0137	3.37	3.41	1.0202	4.94	5.04
1.0008	0.20	0.20	1.0073	1.80	1.82	1.0138	3.39	3.44	1.0203	4.96	5.06
1.0009	0.22	0.22	1.0074	1.83	1.84	1.0139	3.42	3.46	1.0204	4.99	5.09
1.0010	0.25	0.25	1.0075	1.85	1.87	1.0140	3.44	3.49	1.0205	5.01	5.11
1.0011	0.27	0.27	1.0076	1.88	1.89	1.0141	3.46	3.51	1.0206	5.03	5.14
1.0012	0.30	0.30	1.0077	1.90	1.92	1.0142	3.49	3.54	1.0207	5.06	5.16
1.0013	0.32	0.32	1.0078	1.93	1.94	1.0143	3.51	3.56	1.0208	5.08	5.19
1.0014	0.35	0.35	1.0079	1.95	1.97	1.0144	3.54	3.59	1.0209	5.11	5.21
1.0015	0.37	0.37	1.0080	1.98	1.99	1.0145	3.56	3.61	1.0210	5.13	5.24
1.0016	0.40	0.40	1.0081	2.00	2.02	1.0146	3.59	3.64	1.0211	5.15	5.26
1.0017	0.42	0.42	1.0082	2.03	2.04	1.0147	3.61	3.66	1.0212	5.18	5.29
1.0018	0.45	0.45	1.0083	2.05	2.07	1.0148	3.63	3.69	1.0213	5.20	5.31
1.0019	0.47	0.47	1.0084	2.07	2.09	1.0149	3.66	3.71	1.0214	5.23	5.34
1.0020	0.50	0.50	1.0085	2.10	2.12	1.0150	3.68	3.74	1.0215	5.25	5.36
1.0021	0.52	0.52	1.0086	2.12	2.14	1.0151	3.71	3.76	1.0216	5.27	5.39
1.0022	0.55	0.55	1.0087	2.15	2.17	1.0152	3.73	3.79	1.0217	5.30	5.41
1.0023	0.57	0.57	1.0088	2.17	2.19	1.0153	3.76	3.81	1.0218	5.32	5.44
1.0024	0.60	0.60	1.0089	2.20	2.22	1.0154	3.78	3.84	1.0219	5.35	5.46
1.0025	0.62	0.62	1.0090	2.22	2.24	1.0155	3.80	3.86	1.0220	5.37	5.49
1.0026	0.65	0.65	1.0091	2.25	2.27	1.0156	3.83	3.89	1.0221	5.39	5.51
1.0027	0.67	0.67	1.0092	2.27	2.29	1.0157	3.85	3.91	1.0222	5.42	5.54
1.0028	0.69	0.70	1.0093	2.29	2.32	1.0158	3.88	3.94	1.0223	5.44	5.56
1.0029	0.72	0.72	1.0094	2.32	2.34	1.0159	3.90	3.96	1.0224	5.47	5.59
1.0030	0.74	0.75	1.0095	2.34	2.37	1.0160	3.93	3.99	1.0225	5.49	5.61
1.0031	0.77	0.77	1.0096	2.37	2.39	1.0161	3.95	4.01	1.0226	5.51	5.64
1.0032	0.79	0.80	1.0097	2.39	2.42	1.0162	3.97	4.04	1.0227	5.54	5.66
1.0033	0.82	0.82	1.0098	2.42	2.44	1.0163	4.00	4.06	1.0228	5.56	5.69
1.0034	0.84	0.85	1.0099	2.44	2.47	1.0164	4.02	4.09	1.0229	5.59	5.71
1.0035	0.87	0.87	1.0100	2.47	2.49	1.0165	4.05	4.11	1.0230	5.61	5.74
1.0036	0.89	0.90	1.0101	2.49	2.52	1.0166	4.07	4.14	1.0231	5.63	5.76
1.0037	0.92	0.92	1.0102	2.51	2.54	1.0167	4.09	4.16	1.0232	5.66	5.79
1.0038	0.94	0.95	1.0103	2.54	2.57	1.0168	4.12	4.19	1.0233	5.68	5.81
1.0039	0.97	0.97	1.0104	2.56	2.59	1.0169	4.14	4.21	1.0234	5.70	5.84
1.0040	0.99	1.00	1.0105	2.59	2.62	1.0170	4.17	4.24	1.0235	5.73	5.86
1.0041	1.02	1.02	1.0106	2.61	2.64	1.0171	4.19	4.26	1.0236	5.75	5.89
1.0042	1.04	1.05	1.0107	2.64	2.67	1.0172	4.22	4.29	1.0237	5.78	5.91
1.0043	1.07	1.07	1.0108	2.66	2.69	1.0173	4.24	4.31	1.0238	5.80	5.94
1.0044	1.09	1.10	1.0109	2.69	2.72	1.0174	4.26	4.34	1.0239	5.82	5.96
1.0045	1.12	1.12	1.0110	2.71	2.74	1.0175	4.29	4.36	1.0240	5.85	5.99
1.0046	1.14	1.14	1.0111	2.73	2.77	1.0176	4.31	4.39	1.0241	5.87	6.01
1.0047	1.16	1.17	1.0112	2.76	2.79	1.0177	4.34	4.41	1.0242	5.90	6.04
1.0048	1.19	1.19	1.0113	2.78	2.81	1.0178	4.36	4.44	1.0243	5.92	6.06
1.0049	1.21	1.22	1.0114	2.81	2.84	1.0179	4.38	4.46	1.0244	5.94	6.09
1.0050	1.24	1.24	1.0115	2.83	2.86	1.0180	4.41	4.49	1.0245	5.97	6.11
1.0051	1.26	1.27	1.0116	2.86	2.89	1.0181	4.43	4.51	1.0246	5.99	6.14
1.0052	1.29	1.29	1.0117	2.88	2.91	1.0182	4.46	4.54	1.0247	6.01	6.16
1.0053	1.31	1.32	1.0118	2.91	2.94	1.0183	4.48	4.56	1.0248	6.04	6.19
1.0054	1.34	1.34	1.0119	2.93	2.96	1.0184	4.50	4.59	1.0249	6.06	6.21
1.0055	1.36	1.37	1.0120	2.95	2.99	1.0185	4.53	4.61	1.0250	6.09	6.24
1.0056	1.39	1.39	1.0121	2.98	3.01	1.0186	4.55	4.64	1.0251	6.11	6.26
1.0057	1.41	1.42	1.0122	3.00	3.04	1.0187	4.58	4.66	1.0252	6.13	6.29
1.0058	1.44	1.44	1.0123	3.03	3.06	1.0188	4.60	4.69	1.0253	6.16	6.31
1.0059	1.46	1.47	1.0124	3.05	3.09	1.0189	4.63	4.71	1.0254	6.18	6.34
1.0060	1.48	1.49	1.0125	3.08	3.11	1.0190	4.65	4.74	1.0255	6.21	6.36
1.0061	1.51	1.52	1.0126	3.10	3.14	1.0191	4.67	4.76	1.0256	6.23	6.39
1.0062	1.53	1.54	1.0127	3.12	3.16	1.0192	4.70	4.79	1.0257	6.25	6.41
1.0063	1.57	1.56	1.0128	3.15	3.19	1.0193	4.72	4.81	1.0258	6.28	6.44
1.0064	1.58	1.59	1.0129	3.17	3.21	1.0194	4.75	4.84	1.0259	6.30	6.46
1.0065	1.61	1.62	1.0130	3.20	3.24	1.0195	4.77	4.86	1.0260	6.32	6.49

<sup>a</sup> Calculated from results obtained by drying at 97° C.

TABLE IV.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0261	6.35	6.51	1.0326	7.89	8.14	1.0391	9.41	9.77	1.0456	10.91	11.41
1.0262	6.37	6.54	1.0327	7.91	8.17	1.0392	9.43	9.80	1.0457	10.93	11.43
1.0263	6.40	6.56	1.0328	7.93	8.19	1.0393	9.45	9.82	1.0458	10.96	11.46
1.0264	6.42	6.59	1.0329	7.96	8.22	1.0394	9.48	9.85	1.0459	10.98	11.48
1.0265	6.44	6.61	1.0330	7.98	8.24	1.0395	9.50	9.87	1.0460	11.00	11.51
1.0266	6.47	6.64	1.0331	8.00	8.27	1.0396	9.52	9.90	1.0461	11.03	11.53
1.0267	6.49	6.66	1.0332	8.03	8.29	1.0397	9.55	9.92	1.0462	11.05	11.56
1.0268	6.51	6.69	1.0333	8.05	8.32	1.0398	9.57	9.95	1.0463	11.07	11.58
1.0269	6.54	6.71	1.0334	8.07	8.34	1.0399	9.59	9.97	1.0464	11.09	11.61
1.0270	6.56	6.74	1.0335	8.10	8.37	1.0400	9.62	10.00	1.0465	11.12	11.63
1.0271	6.59	6.76	1.0336	8.12	8.39	1.0401	9.64	10.03	1.0466	11.14	11.66
1.0272	6.61	6.79	1.0337	8.14	8.42	1.0402	9.66	10.05	1.0467	11.16	11.68
1.0273	6.63	6.81	1.0338	8.17	8.44	1.0403	9.69	10.08	1.0468	11.19	11.71
1.0274	6.66	6.84	1.0339	8.19	8.47	1.0404	9.71	10.10	1.0469	11.21	11.74
1.0275	6.68	6.86	1.0340	8.21	8.49	1.0405	9.73	10.13	1.0470	11.23	11.76
1.0276	6.70	6.89	1.0341	8.24	8.52	1.0406	9.75	10.15	1.0471	11.26	11.79
1.0277	6.73	6.91	1.0342	8.56	8.54	1.0407	9.78	10.18	1.0472	11.28	11.81
1.0278	6.75	6.94	1.0343	8.28	8.57	1.0408	9.80	10.20	1.0473	11.30	11.84
1.0279	6.78	6.96	1.0344	8.31	8.59	1.0409	9.82	10.23	1.0474	11.32	11.86
1.0280	6.80	6.99	1.0345	8.33	8.62	1.0410	9.85	10.25	1.0475	11.35	11.89
1.0281	6.82	7.01	1.0346	8.36	8.64	1.0411	9.87	10.28	1.0476	11.37	11.91
1.0282	6.85	7.04	1.0347	8.38	8.67	1.0412	9.89	10.30	1.0477	11.39	11.94
1.0283	6.87	7.06	1.0348	8.40	8.69	1.0413	9.92	10.33	1.0478	11.42	11.96
1.0284	6.89	7.09	1.0349	8.43	8.72	1.0414	9.94	10.35	1.0479	11.44	11.99
1.0285	6.92	7.11	1.0350	8.45	8.74	1.0415	9.96	10.38	1.0480	11.46	12.01
1.0286	6.94	7.14	1.0351	8.47	8.77	1.0416	9.99	10.40	1.0481	11.48	12.04
1.0287	6.97	7.16	1.0352	8.50	8.79	1.0417	10.01	10.43	1.0482	11.51	12.06
1.0288	6.99	7.19	1.0353	8.52	8.82	1.0418	10.03	10.45	1.0483	11.53	12.09
1.0289	7.01	7.22	1.0354	8.54	8.84	1.0419	10.06	10.48	1.0484	11.55	12.11
1.0290	7.04	7.24	1.0355	8.57	8.87	1.0420	10.08	10.50	1.0485	11.58	12.14
1.0291	7.06	7.27	1.0356	8.59	8.90	1.0421	10.10	10.53	1.0486	11.60	12.16
1.0292	7.08	7.29	1.0357	8.61	8.92	1.0422	10.13	10.55	1.0487	11.62	12.19
1.0293	7.11	7.32	1.0358	8.64	8.95	1.0423	10.15	10.58	1.0488	11.65	12.21
1.0294	7.13	7.34	1.0359	8.66	8.97	1.0424	10.17	10.60	1.0489	11.67	12.24
1.0295	7.15	7.37	1.0360	8.68	9.00	1.0425	10.20	10.63	1.0490	11.69	12.26
1.0296	7.18	7.39	1.0361	8.71	9.02	1.0426	10.22	10.65	1.0491	11.71	12.29
1.0297	7.20	7.42	1.0362	8.73	9.05	1.0427	10.24	10.68	1.0492	11.74	12.31
1.0298	7.23	7.44	1.0363	8.75	9.07	1.0428	10.26	10.70	1.0493	11.76	12.34
1.0299	7.25	7.47	1.0364	8.78	9.10	1.0429	10.29	10.73	1.0494	11.78	12.36
1.0300	7.27	7.49	1.0365	8.80	9.12	1.0430	10.31	10.75	1.0495	11.81	12.39
1.0301	7.30	7.52	1.0366	8.82	9.15	1.0431	10.33	10.78	1.0496	11.83	12.42
1.0302	7.32	7.54	1.0367	8.85	9.17	1.0432	10.36	10.80	1.0497	11.85	12.44
1.0303	7.34	7.57	1.0368	8.87	9.20	1.0433	10.38	10.83	1.0498	11.87	12.47
1.0304	7.37	7.59	1.0369	8.89	9.22	1.0434	10.40	10.85	1.0499	11.90	12.49
1.0305	7.39	7.62	1.0370	8.92	9.25	1.0435	10.43	10.88	1.0500	11.92	12.52
1.0306	7.41	7.64	1.0371	8.94	9.27	1.0436	10.45	10.90	1.0501	11.94	12.54
1.0307	7.44	7.67	1.0372	8.96	9.30	1.0437	10.47	10.93	1.0502	11.97	12.57
1.0308	7.46	7.69	1.0373	8.99	9.32	1.0438	10.50	10.96	1.0503	11.99	12.59
1.0309	7.49	7.72	1.0374	9.01	9.35	1.0439	10.52	10.98	1.0504	12.01	12.62
1.0310	7.51	7.74	1.0375	9.03	9.37	1.0440	10.54	11.01	1.0505	12.03	12.64
1.0311	7.53	7.77	1.0376	9.06	9.40	1.0441	10.56	11.03	1.0506	12.06	12.67
1.0312	7.56	7.79	1.0377	9.08	9.42	1.0442	10.59	11.06	1.0507	12.08	12.69
1.0313	7.58	7.82	1.0378	9.10	9.45	1.0443	10.61	11.08	1.0508	12.10	12.72
1.0314	7.60	7.84	1.0379	9.13	9.47	1.0444	10.63	11.11	1.0509	12.13	12.74
1.0315	7.63	7.87	1.0380	9.15	9.50	1.0445	10.66	11.13	1.0510	12.15	12.77
1.0316	7.65	7.89	1.0381	9.17	9.52	1.0446	10.68	11.16	1.0511	12.17	12.79
1.0317	7.67	7.92	1.0382	9.20	9.55	1.0447	10.70	11.18	1.0512	12.19	12.82
1.0318	7.70	7.94	1.0383	9.22	9.57	1.0448	10.73	11.21	1.0513	12.22	12.84
1.0319	7.72	7.97	1.0384	9.24	9.60	1.0449	10.75	11.23	1.0514	12.24	12.87
1.0320	7.74	7.99	1.0385	9.27	9.62	1.0450	10.77	11.26	1.0515	12.26	12.89
1.0321	7.77	8.02	1.0386	9.29	9.65	1.0451	10.80	11.28	1.0516	12.28	12.92
1.0322	7.79	8.04	1.0387	9.31	9.67	1.0452	10.82	11.31	1.0517	12.31	12.94
1.0323	7.81	8.07	1.0388	9.34	9.70	1.0453	10.84	11.33	1.0518	12.33	12.97
1.0324	7.84	8.09	1.0389	9.36	9.72	1.0454	10.86	11.36	1.0519	12.35	12.99
1.0325	7.86	8.12	1.0390	9.38	9.75	1.0455	10.88	11.38	1.0520	12.38	13.02

TABLE IV.—*Extract in beer wort*.—Continued.

Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0521	12.40	13.04	1.0586	13.87	14.68	1.0651	15.33	16.33	1.0716	16.77	17.97
1.0522	12.42	13.07	1.0587	13.89	14.71	1.0652	15.35	16.35	1.0717	16.97	18.00
1.0523	12.44	13.10	1.0588	13.92	14.73	1.0653	15.37	16.38	1.0718	16.82	18.02
1.0524	12.47	13.12	1.0589	13.94	14.76	1.0654	15.40	16.40	1.0719	16.84	18.05
1.0525	12.49	13.15	1.0590	13.96	14.79	1.0655	15.42	16.43	1.0720	16.86	18.07
1.0526	12.51	13.17	1.0591	13.98	14.81	1.0656	15.44	16.45	1.0721	16.88	18.10
1.0527	12.54	13.20	1.0592	14.01	14.84	1.0657	15.46	16.48	1.0722	16.90	18.12
1.0528	12.56	13.22	1.0593	14.03	14.86	1.0658	15.48	16.50	1.0723	16.93	18.15
1.0529	12.58	13.25	1.0594	14.05	14.89	1.0659	15.51	16.53	1.0724	16.95	18.17
1.0530	12.60	13.27	1.0595	14.07	14.91	1.0660	15.53	16.55	1.0725	16.97	18.20
1.0531	12.63	13.30	1.0596	14.10	14.94	1.0661	15.55	16.58	1.0726	16.99	18.23
1.0532	12.65	13.32	1.0597	14.12	14.96	1.0662	15.57	16.60	1.0727	17.01	18.25
1.0533	12.67	13.35	1.0598	14.14	14.99	1.0663	15.60	16.63	1.0728	17.04	18.28
1.0534	12.69	13.37	1.0599	14.16	15.01	1.0664	15.62	16.66	1.0729	17.06	18.30
1.0535	12.72	13.40	1.0600	14.19	15.04	1.0665	15.64	16.68	1.0730	17.08	18.33
1.0536	12.74	13.42	1.0601	14.21	15.06	1.0666	15.66	16.71	1.0731	17.10	18.35
1.0537	12.76	13.45	1.0602	14.23	15.09	1.0667	15.69	16.73	1.0732	17.12	18.38
1.0538	12.79	13.47	1.0603	14.25	15.11	1.0668	15.71	16.76	1.0733	17.15	18.40
1.0539	12.81	13.50	1.0604	14.28	15.14	1.0669	15.73	16.78	1.0734	17.17	18.43
1.0540	12.83	13.52	1.0605	14.30	15.16	1.0670	15.75	16.81	1.0735	17.19	18.45
1.0541	12.85	13.55	1.0606	14.32	15.19	1.0671	15.77	16.83	1.0736	17.21	18.48
1.0542	12.88	13.57	1.0607	14.34	15.21	1.0672	15.80	16.86	1.0737	17.23	18.50
1.0543	12.90	13.60	1.0608	14.37	15.24	1.0673	15.82	16.88	1.0738	17.26	18.53
1.0544	12.92	13.62	1.0609	14.39	15.27	1.0674	15.84	16.91	1.0739	17.28	18.55
1.0545	12.94	13.65	1.0610	14.41	15.29	1.0675	15.86	16.93	1.0740	17.30	18.58
1.0546	12.97	13.68	1.0611	14.43	15.32	1.0676	15.89	16.96	1.0741	17.32	18.61
1.0547	12.99	13.70	1.0612	14.46	15.34	1.0677	15.91	16.98	1.0742	17.34	18.63
1.0548	13.01	13.73	1.0613	14.48	15.37	1.0678	15.93	17.01	1.0743	17.37	18.66
1.0549	13.04	13.75	1.0614	14.50	15.39	1.0679	15.95	17.03	1.0744	17.39	18.68
1.0550	13.06	13.78	1.0615	14.52	15.42	1.0680	15.97	17.06	1.0745	17.41	18.71
1.0551	13.08	13.80	1.0616	14.55	15.44	1.0681	16.00	17.09	1.0746	17.43	18.73
1.0552	13.10	13.83	1.0617	14.57	15.47	1.0682	16.02	17.11	1.0747	17.45	18.76
1.0553	13.13	13.85	1.0618	14.59	15.49	1.0683	16.04	17.14	1.0748	17.48	18.78
1.0554	13.15	13.88	1.0619	14.61	15.52	1.0684	16.06	17.16	1.0749	17.50	18.81
1.0555	13.17	13.90	1.0620	14.64	15.54	1.0685	16.09	17.19	1.0750	17.52	18.83
1.0556	13.19	13.93	1.0621	14.66	15.57	1.0686	16.11	17.21	1.0751	17.54	18.86
1.0557	13.22	13.95	1.0622	14.68	15.59	1.0687	16.13	17.24	1.0752	17.56	18.88
1.0558	13.24	13.98	1.0623	14.70	15.62	1.0688	16.15	17.26	1.0753	17.59	18.91
1.0559	13.26	14.00	1.0624	14.73	15.64	1.0689	16.17	17.29	1.0754	17.61	18.93
1.0560	13.28	14.03	1.0625	14.75	15.67	1.0690	16.20	17.31	1.0755	17.63	18.96
1.0561	13.31	14.05	1.0626	14.77	15.69	1.0691	16.22	17.34	1.0756	17.65	18.99
1.0562	13.33	14.08	1.0627	14.79	15.72	1.0692	16.24	17.36	1.0757	17.67	19.01
1.0563	13.35	14.10	1.0628	14.81	15.75	1.0693	16.26	17.39	1.0758	17.69	19.04
1.0564	13.37	14.13	1.0629	14.84	15.77	1.0694	16.28	17.41	1.0759	17.72	19.06
1.0565	13.40	14.15	1.0630	14.86	15.80	1.0695	16.31	17.44	1.0760	17.74	19.09
1.0566	13.42	14.18	1.0631	14.88	15.82	1.0696	16.33	17.47	1.0761	17.76	19.11
1.0567	13.44	14.20	1.0632	14.90	15.85	1.0697	16.35	17.49	1.0762	17.78	19.14
1.0568	13.47	14.23	1.0633	14.93	15.87	1.0698	16.37	17.52	1.0763	17.80	19.16
1.0569	13.49	14.26	1.0634	14.95	15.90	1.0699	16.40	17.54	1.0764	17.83	19.19
1.0570	13.51	14.28	1.0635	14.97	15.92	1.0700	16.42	17.57	1.0765	17.85	19.21
1.0571	13.53	14.31	1.0636	14.99	15.95	1.0701	16.44	17.59	1.0766	17.87	19.24
1.0572	13.56	14.33	1.0637	15.02	15.97	1.0702	16.46	17.62	1.0767	17.89	19.26
1.0573	13.58	14.36	1.0638	15.04	16.00	1.0703	16.48	17.64	1.0768	17.91	19.29
1.0574	13.60	14.38	1.0639	15.06	16.02	1.0704	16.51	17.67	1.0769	17.94	19.32
1.0575	13.62	14.41	1.0640	15.08	16.05	1.0705	16.53	17.69	1.0770	17.96	19.34
1.0576	13.65	14.43	1.0641	15.11	16.07	1.0706	16.55	17.72	1.0771	17.98	19.37
1.0577	13.67	14.46	1.0642	15.13	16.10	1.0707	16.57	17.74	1.0772	18.00	19.39
1.0578	13.69	14.48	1.0643	15.15	16.12	1.0708	16.59	17.77	1.0773	18.02	19.42
1.0579	13.71	14.51	1.0644	15.17	16.15	1.0709	16.62	17.79	1.0774	18.05	19.44
1.0580	13.74	14.53	1.0645	15.19	16.18	1.0710	16.64	17.82	1.0775	18.07	19.47
1.0581	13.76	14.56	1.0646	15.22	16.20	1.0711	16.66	17.85	1.0776	18.09	19.49
1.0582	13.78	14.58	1.0647	15.24	16.23	1.0712	16.68	17.87	1.0777	18.11	19.52
1.0583	13.80	14.61	1.0648	15.26	16.25	1.0713	16.70	17.90	1.0778	18.13	19.54
1.0584	13.83	14.63	1.0649	15.28	16.28	1.0714	16.73	17.92	1.0779	18.15	19.57
1.0585	13.85	14.66	1.0650	15.31	16.30	1.0715	16.75	17.95	1.0780	18.18	19.59

TABLE IV.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0781	18.20	19.62	1.0836	19.39	21.02	1.0891	20.58	22.41	1.0946	21.76	23.81
1.0782	18.22	19.64	1.0837	19.42	21.04	1.0892	20.60	22.44	1.0947	21.78	23.84
1.0783	18.24	19.67	1.0838	19.44	21.07	1.0893	20.62	22.47	1.0948	21.80	23.87
1.0784	18.26	19.70	1.0839	19.46	21.09	1.0894	20.65	22.49	1.0949	21.82	23.89
1.0785	18.29	19.72	1.0840	19.48	21.12	1.0895	20.67	22.52	1.0950	21.84	23.92
1.0786	18.31	19.75	1.0841	19.50	21.14	1.0896	20.69	22.54	1.0951	21.86	23.94
1.0787	18.33	19.77	1.0842	19.52	21.17	1.0897	20.71	22.57	1.0952	21.88	23.97
1.0788	18.35	19.80	1.0843	19.55	21.19	1.0898	20.73	22.59	1.0953	21.91	23.99
1.0789	18.37	19.82	1.0844	19.57	21.22	1.0899	20.75	22.62	1.0954	21.93	24.02
1.0790	18.39	19.85	1.0845	19.59	21.24	1.0900	20.77	22.64	1.0955	21.95	24.04
1.0791	18.42	19.87	1.0846	19.61	21.27	1.0901	20.79	22.67	1.0956	21.97	24.07
1.0792	18.44	19.90	1.0847	19.63	21.30	1.0902	20.82	22.69	1.0957	21.99	24.09
1.0793	18.46	19.92	1.0848	19.65	21.32	1.0903	20.84	22.72	1.0958	22.01	24.12
1.0794	18.48	19.95	1.0849	19.68	21.35	1.0904	20.86	22.75	1.0959	22.03	24.15
1.0795	18.50	19.97	1.0850	19.70	21.37	1.0905	20.88	22.77	1.0960	22.05	24.17
1.0796	18.53	20.00	1.0851	19.72	21.40	1.0906	20.90	22.80	1.0961	22.08	24.20
1.0797	18.55	20.03	1.0852	19.74	21.42	1.0907	20.92	22.82	1.0962	22.10	24.22
1.0798	18.57	20.05	1.0853	19.76	21.45	1.0908	20.94	22.85	1.0963	22.12	24.25
1.0799	18.59	20.08	1.0854	19.78	21.47	1.0909	20.97	22.87	1.0964	22.14	24.27
1.0800	18.61	20.10	1.0855	19.81	21.50	1.0910	20.99	22.90	1.0965	22.16	24.30
1.0801	18.63	20.13	1.0856	19.83	21.52	1.0911	21.01	22.92	1.0966	22.18	24.32
1.0802	18.66	20.15	1.0857	19.85	21.55	1.0912	21.03	22.95	1.0967	22.20	24.35
1.0803	18.68	20.18	1.0858	19.87	21.58	1.0913	21.05	22.97	1.0968	22.22	24.38
1.0804	18.70	20.20	1.0859	19.89	21.60	1.0914	21.07	23.00	1.0969	22.25	24.40
1.0805	18.72	20.23	1.0860	19.91	21.63	1.0915	21.09	23.02	1.0970	22.27	24.43
1.0806	18.74	20.25	1.0861	19.93	21.65	1.0916	21.12	23.05	1.0971	22.29	24.45
1.0807	18.77	20.28	1.0862	19.96	21.68	1.0917	21.14	23.08	1.0972	22.31	24.48
1.0808	18.79	20.30	1.0863	19.98	21.70	1.0918	21.16	23.10	1.0973	22.33	24.50
1.0809	18.81	20.33	1.0864	20.00	21.73	1.0919	21.18	23.13	1.0974	22.35	24.53
1.0810	18.83	20.36	1.0865	20.02	21.75	1.0920	21.20	23.15	1.0975	22.37	24.55
1.0811	18.85	20.38	1.0866	20.04	21.78	1.0921	21.22	23.18	1.0976	22.39	24.58
1.0812	18.87	20.41	1.0867	20.06	21.80	1.0922	21.24	23.20	1.0977	22.41	24.60
1.0813	18.90	20.43	1.0868	20.09	21.83	1.0923	21.27	23.23	1.0978	22.44	24.63
1.0814	18.92	20.46	1.0869	20.11	21.85	1.0924	21.29	23.25	1.0979	22.46	24.66
1.0815	18.94	20.48	1.0870	20.13	21.88	1.0925	21.31	33.28	1.0980	22.48	24.68
1.0816	18.96	20.51	1.0871	20.15	21.91	1.0926	21.33	23.31	1.0981	22.50	24.71
1.0817	18.98	20.53	1.0872	20.17	21.93	1.0927	21.35	23.33	1.0982	22.52	24.73
1.0818	19.00	20.56	1.0873	20.19	21.96	1.0928	21.37	23.36	1.0983	22.54	24.76
1.0819	19.03	20.58	1.0874	25.21	21.98	1.0929	21.39	23.38	1.0984	22.56	25.78
1.0820	19.05	20.61	1.0875	20.24	22.01	1.0930	21.42	23.41	1.0985	22.58	24.81
1.0821	19.07	20.63	1.0876	20.26	22.03	1.0931	21.44	23.43	1.0986	22.61	24.83
1.0822	19.09	20.66	1.0877	20.28	22.06	1.0932	21.46	23.46	1.0987	22.63	24.86
1.0823	19.11	20.69	1.0878	20.30	22.08	1.0933	21.48	23.48	1.0988	22.65	24.89
1.0824	19.13	20.71	1.0879	20.32	22.11	1.0934	21.50	23.51	1.0989	22.67	24.91
1.0825	19.16	20.74	1.0880	20.34	22.13	1.0935	11.52	23.53	1.0990	22.69	24.94
1.0826	19.18	20.76	1.0881	20.37	22.16	1.0936	21.54	23.56	1.0991	22.71	24.96
1.0827	19.20	20.79	1.0882	10.39	22.19	1.0937	21.56	23.59	1.0992	22.73	24.99
1.0828	19.22	20.81	1.0883	20.41	22.21	1.0938	21.59	23.61			
1.0829	19.24	20.84	1.0884	20.43	22.24	1.0939	21.61	23.64			
1.0830	19.26	20.86	1.0885	20.45	22.26	1.0940	21.63	23.66			
1.0831	19.29	20.89	1.0886	20.47	22.29	1.0941	21.65	23.69			
1.0832	19.31	20.91	1.0887	20.49	22.31	1.0942	21.67	23.71			
1.0833	19.33	20.94	1.0888	20.52	22.34	1.0943	21.69	23.74			
1.0834	19.35	20.96	1.0889	20.54	22.36	1.0944	21.71	23.76			
1.0835	19.37	20.99	1.0890	20.56	22.39	1.0945	21.73	23.79			

TABLE V.—*Extract in wine.*

[According to Windisch.]

Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.
1.0000	0.00	1.0065	1.68	1.0130	3.36	1.0195	5.04	1.0260	6.72	1.0325	8.40
1.0001	0.03	1.0066	1.70	1.0131	3.38	1.0196	5.06	1.0261	6.75	1.0326	8.43
1.0002	0.05	1.0067	1.73	1.0132	3.41	1.0197	5.09	1.0262	6.77	1.0327	8.46
1.0003	0.08	1.0068	1.76	1.0133	3.43	1.0198	5.11	1.0263	6.80	1.0328	8.48
1.0004	0.10	1.0069	1.78	1.0134	3.46	1.0199	5.14	1.0264	6.82	1.0329	8.51
1.0005	0.13	1.0070	1.81	1.0135	3.49	1.0200	5.17	1.0265	6.85	1.0330	8.53
1.0006	0.15	1.0071	1.83	1.0136	3.51	1.0201	5.19	1.0266	6.88	1.0331	8.56
1.0007	0.18	1.0072	1.86	1.0137	3.54	1.0202	5.22	1.0267	6.90	1.0332	8.59
1.0008	0.20	1.0073	1.88	1.0138	3.56	1.0203	5.25	1.0268	6.93	1.0333	8.61
1.0009	0.23	1.0074	1.91	1.0139	3.59	1.0204	5.27	1.0269	6.95	1.0334	8.64
1.0010	0.26	1.0075	1.94	1.0140	3.62	1.0205	5.30	1.0270	6.98	1.0335	8.66
1.0011	0.28	1.0076	1.96	1.0141	3.64	1.0206	5.32	1.0271	7.01	1.0336	8.69
1.0012	0.31	1.0077	1.99	1.0142	3.67	1.0207	5.35	1.0272	7.03	1.0337	8.72
1.0013	0.34	1.0078	2.01	1.0143	3.69	1.0208	5.38	1.0273	7.06	1.0338	8.74
1.0014	0.36	1.0079	2.04	1.0144	3.72	1.0209	5.40	1.0274	7.08	1.0339	8.77
1.0015	0.39	1.0080	2.07	1.0145	3.75	1.0210	5.43	1.0275	7.11	1.0340	8.79
1.0016	0.41	1.0081	2.09	1.0146	3.77	1.0211	5.45	1.0276	7.13	1.0341	8.82
1.0017	0.44	1.0082	2.12	1.0147	3.80	1.0212	5.48	1.0277	7.16	1.0342	8.85
1.0018	0.46	1.0083	2.14	1.0148	3.82	1.0213	5.51	1.0278	7.19	1.0343	8.87
1.0019	0.49	1.0084	2.17	1.0149	3.85	1.0214	5.53	1.0279	7.21	1.0344	8.90
1.0020	0.52	1.0085	2.19	1.0150	3.87	1.0215	5.56	1.0280	7.24	1.0345	8.92
1.0021	0.54	1.0086	2.22	1.0151	3.90	1.0216	5.58	1.0281	7.26	1.0346	8.95
1.0022	0.57	1.0087	2.25	1.0152	3.93	1.0217	5.61	1.0282	7.29	1.0347	8.97
1.0023	0.59	1.0088	2.27	1.0153	3.95	1.0218	5.64	1.0283	7.32	1.0348	9.00
1.0024	0.62	1.0089	2.30	1.0154	3.98	1.0219	5.66	1.0284	7.34	1.0349	9.03
1.0025	0.64	1.0090	2.32	1.0155	4.00	1.0220	5.69	1.0285	7.37	1.0350	9.05
1.0026	0.67	1.0091	2.35	1.0156	4.03	1.0221	5.71	1.0286	7.39	1.0351	9.08
1.0027	0.69	1.0092	2.38	1.0157	4.06	1.0222	5.74	1.0287	7.42	1.0352	9.10
1.0028	0.72	1.0093	2.40	1.0158	4.08	1.0223	5.77	1.0288	7.45	1.0353	9.13
1.0029	0.75	1.0094	2.43	1.0159	4.11	1.0224	5.79	1.0289	7.47	1.0354	9.16
1.0030	0.77	1.0095	2.45	1.0160	4.13	1.0225	5.82	1.0290	7.50	1.0355	9.18
1.0031	0.80	1.0096	2.48	1.0161	4.16	1.0226	5.84	1.0291	7.52	1.0356	9.21
1.0032	0.82	1.0097	2.50	1.0162	4.19	1.0227	5.87	1.0292	7.55	1.0357	9.23
1.0033	0.85	1.0098	2.53	1.0163	4.21	1.0228	5.89	1.0293	7.58	1.0358	9.26
1.0034	0.87	1.0099	2.56	1.0164	4.24	1.0229	5.92	1.0294	7.60	1.0359	9.29
1.0035	0.90	1.0100	2.58	1.0165	4.26	1.0230	5.94	1.0295	7.63	1.0360	9.31
1.0036	0.93	1.0101	2.61	1.0166	4.29	1.0231	5.97	1.0296	7.65	1.0361	9.34
1.0037	0.95	1.0102	2.63	1.0167	4.31	1.0232	6.00	1.0297	7.68	1.0362	9.36
1.0038	0.98	1.0103	2.66	1.0168	4.34	1.0233	6.02	1.0298	7.70	1.0363	9.39
1.0039	1.00	1.0104	2.69	1.0169	4.37	1.0234	6.05	1.0299	7.73	1.0364	9.42
1.0040	1.03	1.0105	2.71	1.0170	4.39	1.0235	6.07	1.0300	7.76	1.0365	9.44
1.0041	1.05	1.0106	2.74	1.0171	4.42	1.0236	6.10	1.0301	7.78	1.0366	9.47
1.0042	1.08	1.0107	2.76	1.0172	4.44	1.0237	6.12	1.0302	7.81	1.0367	9.49
1.0043	1.11	1.0108	2.79	1.0173	4.47	1.0238	6.15	1.0303	7.83	1.0368	9.52
1.0044	1.13	1.0109	2.82	1.0174	4.50	1.0239	6.18	1.0304	7.86	1.0369	9.55
1.0045	1.16	1.0110	2.84	1.0175	4.52	1.0240	6.20	1.0305	7.89	1.0370	9.57
1.0046	1.18	1.0111	2.87	1.0176	4.55	1.0241	6.23	1.0306	7.91	1.0371	9.60
1.0047	1.21	1.0112	2.89	1.0177	4.57	1.0242	6.25	1.0307	7.94	1.0372	9.62
1.0048	1.24	1.0113	2.92	1.0178	4.60	1.0243	6.28	1.0308	7.97	1.0373	9.65
1.0049	1.26	1.0114	2.94	1.0179	4.63	1.0244	6.31	1.0309	7.99	1.0374	9.68
1.0050	1.29	1.0115	2.97	1.0180	4.65	1.0245	6.33	1.0310	8.02	1.0375	9.70
1.0051	1.32	1.0116	3.00	1.0181	4.68	1.0246	6.36	1.0311	8.04	1.0376	9.73
1.0052	1.34	1.0117	3.02	1.0182	4.70	1.0247	6.38	1.0312	8.07	1.0377	9.75
1.0053	1.37	1.0118	3.05	1.0183	4.73	1.0248	6.41	1.0313	8.09	1.0378	9.78
1.0054	1.39	1.0119	3.07	1.0184	4.75	1.0249	6.44	1.0314	8.12	1.0379	9.80
1.0055	1.42	1.0120	3.10	1.0185	4.78	1.0250	6.46	1.0315	8.14	1.0380	9.83
1.0056	1.45	1.0121	3.12	1.0186	4.81	1.0251	6.49	1.0316	8.17	1.0381	9.86
1.0057	1.47	1.0122	3.15	1.0187	4.83	1.0252	6.51	1.0317	8.20	1.0382	9.88
1.0058	1.50	1.0123	3.18	1.0188	4.86	1.0253	6.54	1.0318	8.22	1.0383	9.91
1.0059	1.52	1.0124	3.20	1.0189	4.88	1.0254	6.56	1.0319	8.25	1.0384	9.93
1.0060	1.55	1.0125	3.23	1.0190	4.91	1.0255	6.59	1.0320	8.27	1.0385	9.96
1.0061	1.57	1.0126	3.26	1.0191	4.94	1.0256	6.62	1.0321	8.30	1.0386	9.99
1.0062	1.60	1.0127	3.28	1.0192	4.96	1.0257	6.64	1.0322	8.33	1.0387	10.01
1.0063	1.63	1.0128	3.31	1.0193	4.99	1.0258	6.67	1.0323	8.35	1.0388	10.04
1.0064	1.65	1.0129	3.33	1.0194	5.01	1.0259	6.70	1.0324	8.38	1.0389	10.06



TABLE V.—*Extract in wine*—Continued.

Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.
1.0390	10.09	1.0455	11.78	1.0520	13.47	1.0585	15.16	1.0650	16.86	1.0715	18.56
1.0391	10.11	1.0456	11.81	1.0521	13.49	1.0586	15.19	1.0651	16.88	1.0716	18.58
1.0392	10.14	1.0457	11.83	1.0522	13.52	1.0587	15.22	1.0652	16.91	1.0717	18.61
1.0393	10.17	1.0458	11.86	1.0523	13.55	1.0588	15.24	1.0653	16.94	1.0718	18.63
1.0394	10.19	1.0459	11.88	1.0524	13.57	1.0589	15.27	1.0654	16.96	1.0719	18.66
1.0395	10.22	1.0460	11.91	1.0525	13.60	1.0590	15.29	1.0655	16.99	1.0720	18.69
1.0396	10.25	1.0461	11.94	1.0526	13.62	1.0591	15.32	1.0656	17.01	1.0721	18.71
1.0397	10.27	1.0462	11.96	1.0527	13.65	1.0592	15.35	1.0657	17.04	1.0722	18.74
1.0398	10.30	1.0463	11.99	1.0528	13.68	1.0593	15.37	1.0658	17.07	1.0723	18.76
1.0399	10.32	1.0464	12.01	1.0529	13.70	1.0594	15.40	1.0659	17.09	1.0724	18.79
1.0400	10.35	1.0465	12.04	1.0530	13.73	1.0595	15.42	1.0660	17.12	1.0725	18.82
1.0401	10.37	1.0466	12.06	1.0531	13.75	1.0596	15.45	1.0661	17.14	1.0726	18.84
1.0402	10.40	1.0467	12.09	1.0532	13.78	1.0597	15.48	1.0662	17.17	1.0727	18.87
1.0403	10.43	1.0468	12.12	1.0533	13.81	1.0598	15.50	1.0663	17.20	1.0728	18.90
1.0404	10.45	1.0469	12.14	1.0534	13.83	1.0599	15.53	1.0664	17.22	1.0729	18.92
1.0405	10.48	1.0470	12.17	1.0535	13.86	1.0600	15.55	1.0665	17.25	1.0730	18.95
1.0406	10.51	1.0471	12.19	1.0536	13.89	1.0601	15.58	1.0666	17.27	1.0731	18.97
1.0407	10.53	1.0472	12.22	1.0537	13.91	1.0602	15.61	1.0667	17.30	1.0732	19.00
1.0408	10.56	1.0473	12.25	1.0538	13.94	1.0603	15.63	1.0668	17.33	1.0733	19.03
1.0409	10.58	1.0474	12.27	1.0539	13.96	1.0604	15.66	1.0669	17.35	1.0734	19.05
1.0410	10.61	1.0475	12.30	1.0540	13.99	1.0605	15.68	1.0670	17.38	1.0735	19.08
1.0411	10.63	1.0476	12.32	1.0541	14.01	1.0606	15.71	1.0671	17.41	1.0736	19.10
1.0412	10.66	1.0477	12.35	1.0542	14.04	1.0607	15.74	1.0672	17.43	1.0737	19.13
1.0413	10.69	1.0478	12.38	1.0543	15.07	1.0608	15.76	1.0673	17.46	1.0738	19.16
1.0414	10.71	1.0479	12.40	1.0544	14.09	1.0609	15.79	1.0674	17.48	1.0739	19.18
1.0415	10.74	1.0480	12.43	1.0545	14.12	1.0610	15.81	1.0675	17.51	1.0740	19.21
1.0416	10.76	1.0481	12.45	1.0546	14.14	1.0611	15.84	1.0676	17.54	1.0741	19.23
1.0417	10.79	1.0482	12.48	1.0547	14.17	1.0612	15.87	1.0677	17.56	1.0742	19.26
1.0418	10.82	1.0483	12.51	1.0548	14.20	1.0613	15.89	1.0678	17.59	1.0743	19.29
1.0419	10.84	1.0484	12.53	1.0549	14.22	1.0614	15.92	1.0679	17.62	1.0744	19.31
1.0420	10.87	1.0485	12.56	1.0550	14.25	1.0615	15.94	1.0680	17.64	1.0745	19.34
1.0421	10.90	1.0486	12.58	1.0551	14.28	1.0616	15.97	1.0681	17.67	1.0746	19.37
1.0422	10.92	1.0487	12.61	1.0552	14.30	1.0617	16.00	1.0682	17.69	1.0747	19.39
1.0423	10.95	1.0488	12.64	1.0553	14.33	1.0618	16.02	1.0683	17.72	1.0748	19.42
1.0424	10.97	1.0489	12.66	1.0554	14.35	1.0619	16.05	1.0684	17.75	1.0749	19.44
1.0425	11.00	1.0490	12.69	1.0555	14.38	1.0620	16.07	1.0685	17.77	1.0750	19.47
1.0426	11.03	1.0491	12.71	1.0556	14.41	1.0621	16.10	1.0686	17.80	1.0751	19.50
1.0427	11.05	1.0492	12.74	1.0557	14.43	1.0622	16.13	1.0687	17.83	1.0752	19.52
1.0428	11.08	1.0493	12.77	1.0558	14.46	1.0623	16.15	1.0688	17.85	1.0753	19.55
1.0429	11.10	1.0494	12.79	1.0559	14.48	1.0624	16.18	1.0689	17.88	1.0754	19.58
1.0430	11.13	1.0495	12.82	1.0560	14.51	1.0625	16.21	1.0690	17.90	1.0755	19.60
1.0431	11.15	1.0496	12.84	1.0561	14.54	1.0626	16.23	1.0691	17.93	1.0756	19.63
1.0432	11.18	1.0497	12.87	1.0562	14.56	1.0627	16.26	1.0692	17.95	1.0757	19.65
1.0433	11.21	1.0498	12.90	1.0563	14.59	1.0628	16.28	1.0693	17.98	1.0758	19.68
1.0434	11.23	1.0499	12.92	1.0564	14.61	1.0629	16.31	1.0694	18.01	1.0759	19.71
1.0435	11.26	1.0500	12.95	1.0565	14.64	1.0630	16.33	1.0695	18.03	1.0760	19.73
1.0436	11.28	1.0501	12.97	1.0566	14.67	1.0631	16.36	1.0696	18.06	1.0761	19.76
1.0437	11.31	1.0502	13.00	1.0567	14.69	1.0632	16.39	1.0697	18.08	1.0762	19.79
1.0438	11.34	1.0503	13.03	1.0568	14.72	1.0633	16.41	1.0698	18.11	1.0763	19.81
1.0439	11.36	1.0504	13.05	1.0569	14.74	1.0634	16.44	1.0699	18.14	1.0764	19.84
1.0440	11.39	1.0505	13.08	1.0570	14.77	1.0635	16.47	1.0700	18.16	1.0765	19.86
1.0441	11.42	1.0506	13.10	1.0571	14.80	1.0636	16.49	1.0701	18.19	1.0766	19.89
1.0442	11.44	1.0507	13.13	1.0572	14.82	1.0637	16.52	1.0702	18.22	1.0767	19.92
1.0443	11.47	1.0508	13.16	1.0573	14.85	1.0638	16.54	1.0703	18.24	1.0768	19.94
1.0444	11.49	1.0509	13.18	1.0574	14.87	1.0639	16.57	1.0704	18.27	1.0769	19.97
1.0445	11.52	1.0510	13.21	1.0575	14.90	1.0640	16.60	1.0705	18.30	1.0770	20.00
1.0446	11.55	1.0511	13.23	1.0576	14.93	1.0641	16.62	1.0706	18.32	1.0771	20.02
1.0447	11.57	1.0512	13.26	1.0577	14.95	1.0642	16.65	1.0707	18.35	1.0772	20.05
1.0448	11.60	1.0513	13.29	1.0578	14.98	1.0643	16.68	1.0708	18.37	1.0773	20.07
1.0449	11.62	1.0514	13.31	1.0579	15.00	1.0644	16.70	1.0709	18.40	1.0774	20.10
1.0450	11.65	1.0515	13.34	1.0580	15.03	1.0645	16.73	1.0710	18.43	1.0775	20.12
1.0451	11.68	1.0516	13.36	1.0581	15.06	1.0646	16.75	1.0711	18.45	1.0776	20.15
1.0452	11.70	1.0517	13.39	1.0582	15.08	1.0647	16.78	1.0712	18.48	1.0777	20.18
1.0453	11.73	1.0518	13.42	1.0583	15.11	1.0648	16.80	1.0713	18.50	1.0778	20.20
1.0454	11.75	1.0519	13.44	1.0584	15.14	1.0649	16.83	1.0714	18.53	1.0779	20.23

TABLE V.—*Extract in wine*—Continued.

Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.
1.0780	20.26	1.0845	21.96	1.0910	23.67	1.0975	25.38	1.1040	27.09	1.1105	28.81
1.0781	20.28	1.0846	21.99	1.0911	23.70	1.0976	25.41	1.1041	27.12	1.1106	28.83
1.0782	20.31	1.0847	22.02	1.0912	23.72	1.0977	25.43	1.1042	27.15	1.1107	28.86
1.0783	20.34	1.0848	22.04	1.0913	23.75	1.0978	25.46	1.1043	27.17	1.1108	28.88
1.0784	20.36	1.0849	22.07	1.0914	23.77	1.0979	25.49	1.1044	27.20	1.1109	28.91
1.0785	20.39	1.0850	22.09	1.0915	23.80	1.0980	25.51	1.1045	27.22	1.1110	28.94
1.0786	20.41	1.0851	22.12	1.0916	23.83	1.0981	25.54	1.1046	27.25	1.1111	28.96
1.0787	20.44	1.0852	22.15	1.0917	23.85	1.0982	25.56	1.1047	27.27	1.1112	28.99
1.0788	20.47	1.0853	22.17	1.0918	23.88	1.0983	25.59	1.1048	27.30	1.1113	29.02
1.0789	20.49	1.0854	22.20	1.0919	23.91	1.0984	25.62	1.1049	27.33	1.1114	29.04
1.0790	20.52	1.0855	22.22	1.0920	23.93	1.0985	25.64	1.1050	27.35	1.1115	29.07
1.0791	20.55	1.0856	22.25	1.0921	23.96	1.0986	25.67	1.1051	27.38	1.1116	29.09
1.0792	20.57	1.0857	22.28	1.0922	23.99	1.0987	25.70	1.1052	27.41	1.1117	29.12
1.0793	20.60	1.0858	22.30	1.0923	24.01	1.0988	25.72	1.1053	27.43	1.1118	29.15
1.0794	20.62	1.0859	22.33	1.0924	24.04	1.0989	25.75	1.1054	27.46	1.1119	29.17
1.0795	20.65	1.0860	22.36	1.0925	24.07	1.0990	25.78	1.1055	27.49	1.1120	29.20
1.0796	20.68	1.0861	22.38	1.0926	24.09	1.0991	25.80	1.1056	27.51	1.1121	29.23
1.0797	20.70	1.0862	22.41	1.0927	24.12	1.0992	25.83	1.1057	27.54	1.1122	29.25
1.0798	20.73	1.0863	22.43	1.0928	24.14	1.0993	25.85	1.1058	27.57	1.1123	29.28
1.0799	20.75	1.0864	22.46	1.0929	24.17	1.0994	25.88	1.1059	27.59	1.1124	29.31
1.0800	20.78	1.0865	22.49	1.0930	24.20	1.0995	25.91	1.1060	27.62	1.1125	29.33
1.0801	20.81	1.0866	22.51	1.0931	24.22	1.0996	25.93	1.1061	27.65	1.1126	29.36
1.0802	20.83	1.0867	22.54	1.0932	24.25	1.0997	25.96	1.1062	27.67	1.1127	29.39
1.0803	20.86	1.0868	22.57	1.0933	24.27	1.0998	25.99	1.1063	27.70	1.1128	29.41
1.0804	20.89	1.0869	22.59	1.0934	24.30	1.0999	26.01	1.1064	27.72	1.1129	29.44
1.0805	20.91	1.0870	22.62	1.0935	24.33	1.1000	26.04	1.1065	27.75	1.1130	29.47
1.0806	20.94	1.0871	22.65	1.0936	24.35	1.1001	26.06	1.1066	27.78	1.1131	29.49
1.0807	20.96	1.0872	22.67	1.0937	24.38	1.1002	26.09	1.1067	27.80	1.1132	29.52
1.0808	20.99	1.0873	22.70	1.0938	24.41	1.1003	26.12	1.1068	27.83	1.1133	29.54
1.0809	21.02	1.0874	22.72	1.0939	24.43	1.1004	26.14	1.1069	27.86	1.1134	29.57
1.0810	21.04	1.0875	22.75	1.0940	24.46	1.1005	26.17	1.1070	27.88	1.1135	29.60
1.0811	21.07	1.0876	22.78	1.0941	24.49	1.1006	26.20	1.1071	27.96	1.1136	29.62
1.0812	21.10	1.0877	22.80	1.0942	24.51	1.1007	26.22	1.1072	27.93	1.1137	29.65
1.0813	21.12	1.0878	22.83	1.0943	24.54	1.1008	26.25	1.1073	27.96	1.1138	29.68
1.0814	21.15	1.0879	22.86	1.0944	24.57	1.1009	26.27	1.1074	27.99	1.1139	29.70
1.0815	21.17	1.0880	22.88	1.0945	24.59	1.1010	26.30	1.1075	28.01	1.1140	29.73
1.0816	21.20	1.0881	22.91	1.0946	24.62	1.1011	26.33	1.1076	28.04	1.1141	29.76
1.0817	21.23	1.0882	22.93	1.0947	24.64	1.1012	26.35	1.1077	28.07	1.1142	29.78
1.0818	21.25	1.0883	22.96	1.0948	24.67	1.1013	26.38	1.1078	28.09	1.1143	29.81
1.0819	21.28	1.0884	22.99	1.0949	24.70	1.1014	26.41	1.1079	28.12	1.1144	29.83
1.0820	21.31	1.0885	23.01	1.0950	24.72	1.1015	26.43	1.1080	28.15	1.1145	29.86
1.0821	21.33	1.0886	23.04	1.0951	24.75	1.1016	26.46	1.1081	28.17	1.1146	29.89
1.0822	21.36	1.0887	23.07	1.0952	24.78	1.1017	26.49	1.1082	28.20	1.1147	29.91
1.0823	21.38	1.0888	23.09	1.0953	24.80	1.1018	26.51	1.1083	28.22	1.1148	29.94
1.0824	21.41	1.0889	23.12	1.0954	24.83	1.1019	26.54	1.1084	28.25	1.1149	29.96
1.0825	21.44	1.0890	23.14	1.0955	24.85	1.1020	26.56	1.1085	28.28	1.1150	29.99
1.0826	21.46	1.0891	23.17	1.0956	24.88	1.1021	26.59	1.1086	28.30	1.1151	30.02
1.0827	21.49	1.0892	23.20	1.0957	24.91	1.1022	26.62	1.1087	28.33	1.1152	30.04
1.0828	21.52	1.0893	23.22	1.0958	24.93	1.1023	26.64	1.1088	28.36	1.1153	30.07
1.0829	21.54	1.0894	23.25	1.0959	24.96	1.1024	26.67	1.1089	28.38	1.1154	30.10
1.0830	21.57	1.0895	23.28	1.0960	24.99	1.1025	26.70	1.1090	28.41	1.1155	30.13
1.0831	21.59	1.0896	23.30	1.0961	25.01	1.1026	26.72	1.1091	28.43	1.1156	30.15
1.0832	21.62	1.0897	23.33	1.0962	25.04	1.1027	26.75	1.1092	28.46	1.1157	30.18
1.0833	21.65	1.0898	23.35	1.0963	25.07	1.1028	26.78	1.1093	28.49	1.1158	30.21
1.0834	21.67	1.0899	23.38	1.0964	25.09	1.1029	26.80	1.1094	28.51	1.1159	30.23
1.0835	21.70	1.0900	23.41	1.0965	25.12	1.1030	26.83	1.1095	28.54		
1.0836	21.73	1.0901	23.43	1.0966	25.14	1.1031	26.85	1.1096	28.57		
1.0837	21.75	1.0902	23.46	1.0967	25.17	1.1032	26.88	1.1097	28.59		
1.0838	21.78	1.0903	23.49	1.0968	25.20	1.1033	26.91	1.1098	28.62		
1.0839	21.80	1.0904	23.51	1.0969	25.22	1.1034	26.93	1.1099	28.65		
1.0840	21.83	1.0905	23.54	1.0970	25.25	1.1035	26.96	1.1100	28.67		
1.0841	21.86	1.0906	23.57	1.0971	25.28	1.1036	26.99	1.1101	28.70		
1.0842	21.88	1.0907	23.59	1.0972	25.30	1.1037	27.01	1.1102	28.73		
1.0843	21.91	1.0908	23.62	1.0973	25.33	1.1038	27.04	1.1103	28.75		
1.0844	21.94	1.0909	23.65	1.0974	25.36	1.1039	27.07	1.1104	28.78		

TABLE VI.—*Relation of brix, specific gravity, and Baumé.*

Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.
0.1	1.0003	0.06	6.6	1.0261	3.7	13.1	1.0531	7.3	19.6	1.0815	10.85
0.2	1.0007	0.11	6.7	1.0265	3.7	13.2	1.0536	7.3	19.7	1.0819	10.9
0.3	1.0011	0.17	6.8	1.0269	3.8	13.3	1.0540	7.4	19.8	1.0824	11.0
0.4	1.0015	0.22	6.9	1.0273	3.8	13.4	1.0544	7.4	19.9	1.0828	11.0
0.5	1.0019	0.28	7.0	1.0277	3.9	13.5	1.0548	7.5	20.0	1.0832	11.1
0.6	1.0023	0.33	7.1	1.0281	3.9	13.6	1.0553	7.5	20.1	1.0837	11.1
0.7	1.0027	0.39	7.2	1.0286	4.0	13.7	1.0557	7.6	20.2	1.0841	11.2
0.8	1.0031	0.44	7.3	1.0290	4.1	13.8	1.0561	7.65	20.3	1.0846	11.2
0.9	1.0034	0.5	7.4	1.0294	4.1	13.9	1.0566	7.7	20.4	1.0850	11.3
1.0	1.0038	0.55	7.5	1.0298	4.2	14.0	1.0570	7.8	20.5	1.0855	11.3
1.1	1.0042	0.6	7.6	1.0302	4.2	14.1	1.0574	7.8	20.6	1.0859	11.4
1.2	1.0046	0.7	7.7	1.0306	4.3	14.2	1.0578	7.9	20.7	1.0864	11.45
1.3	1.0050	0.7	7.8	1.0310	4.3	14.3	1.0583	7.9	20.8	1.0868	11.5
1.4	1.0054	0.8	7.9	1.0314	4.4	14.4	1.0587	8.0	20.9	1.0873	11.6
1.5	1.0058	0.8	8.0	1.0318	4.4	14.5	1.0591	8.0	21.0	1.0877	11.6
1.6	1.0062	0.9	8.1	1.0322	4.5	14.6	1.0596	8.1	21.1	1.0882	11.7
1.7	1.0066	0.9	8.2	1.0327	4.55	14.7	1.0600	8.15	21.2	1.0886	11.7
1.8	1.0070	1.0	8.3	1.0331	4.6	14.8	1.0604	8.2	21.3	1.0891	11.8
1.9	1.0074	1.05	8.4	1.0335	4.7	14.9	1.0609	8.3	21.4	1.0895	11.8
2.0	1.0077	1.1	8.5	1.0339	4.7	15.0	1.0613	8.3	21.5	1.0900	11.9
2.1	1.0081	1.2	8.6	1.0343	4.8	15.1	1.0617	8.4	21.6	1.0904	11.95
2.2	1.0085	1.2	8.7	1.0347	4.8	15.2	1.0621	8.4	21.7	1.0909	12.0
2.3	1.0089	1.3	8.8	1.0351	4.9	15.3	1.0626	8.5	21.8	1.0914	12.05
2.4	1.0093	1.3	8.9	1.0355	4.9	15.4	1.0630	8.5	21.9	1.0918	12.1
2.5	1.0097	1.4	9.0	1.0359	5.0	15.5	1.0634	8.6	22.0	1.0923	12.2
2.6	1.0101	1.4	9.1	1.0364	5.05	15.6	1.0639	8.65	22.1	1.0927	12.2
2.7	1.0105	1.5	9.2	1.0368	5.1	15.7	1.0643	8.7	22.2	1.0932	12.3
2.8	1.0109	1.55	9.3	1.0372	5.2	15.8	1.0647	8.8	22.3	1.0936	12.3
2.9	1.0113	1.6	9.4	1.0376	5.2	15.9	1.0652	8.8	22.4	1.0941	12.4
3.0	1.0117	1.7	9.5	1.0380	5.3	16.0	1.0656	8.9	22.5	1.0945	12.4
3.1	1.0121	1.7	9.6	1.0384	5.3	16.1	1.0660	8.9	22.6	1.0950	12.5
3.2	1.0125	1.8	9.7	1.0388	5.4	16.2	1.0665	9.0	22.7	1.0954	12.55
3.3	1.0129	1.8	9.8	1.0393	5.4	16.3	1.0669	9.0	22.8	1.0959	12.6
3.4	1.0133	1.9	9.9	1.0397	5.5	16.4	1.0674	9.1	22.9	1.0964	12.7
3.5	1.0137	1.9	10.0	1.0401	5.55	16.5	1.0678	9.1	23.0	1.0968	12.7
3.6	1.0141	2.0	10.1	1.0405	5.6	16.6	1.0682	9.2	23.1	1.0973	12.8
3.7	1.0145	2.0	10.2	1.0409	5.7	16.7	1.0687	9.25	23.2	1.0977	12.8
3.8	1.0149	2.1	10.3	1.0413	5.7	16.8	1.0691	9.3	23.3	1.0982	12.9
3.9	1.0153	2.2	10.4	1.0418	5.8	16.9	1.0695	9.4	23.4	1.0986	12.9
4.0	1.0157	2.2	10.5	1.0422	5.8	17.0	1.0700	9.4	23.5	1.0991	13.0
4.1	1.0161	2.3	10.6	1.0426	5.9	17.1	1.0704	9.5	23.6	1.0996	13.0
4.2	1.0165	2.3	10.7	1.0430	5.9	17.2	1.0709	9.5	23.7	1.1000	13.1
4.3	1.0169	2.4	10.8	1.0434	6.0	17.3	1.0713	9.6	23.8	1.1005	13.15
4.4	1.0173	2.4	10.9	1.0439	6.05	17.4	1.0717	9.6	23.9	1.1009	13.2
4.5	1.0177	2.5	11.0	1.0443	6.1	17.5	1.0722	9.7	24.0	1.1014	13.3
4.6	1.0181	2.6	11.1	1.0447	6.2	17.6	1.0726	9.75	24.1	1.1019	13.3
4.7	1.0185	2.6	11.2	1.0451	6.2	17.7	1.0730	9.8	24.2	1.1023	13.4
4.8	1.0189	2.7	11.3	1.0455	6.3	17.8	1.0735	9.9	24.3	1.1028	13.4
4.9	1.0193	2.7	11.4	1.0459	6.3	17.9	1.0739	9.9	24.4	1.1032	13.5
5.0	1.0197	2.8	11.5	1.0464	6.4	18.0	1.0744	10.0	24.5	1.1037	13.5
5.1	1.0201	2.8	11.6	1.0468	6.4	18.1	1.0748	10.0	24.6	1.1042	13.6
5.2	1.0205	2.9	11.7	1.0472	6.5	18.2	1.0753	10.1	24.7	1.1046	13.6
5.3	1.0209	2.9	11.8	1.0476	6.55	18.3	1.0757	10.1	24.8	1.1051	13.7
5.4	1.0213	3.0	11.9	1.0481	6.6	18.4	1.0761	10.2	24.9	1.1056	13.75
5.5	1.0217	3.0	12.0	1.0485	6.7	18.5	1.0766	10.2	25.0	1.1060	13.8
5.6	1.0221	3.1	12.1	1.0489	6.7	18.6	1.0770	10.3	25.1	1.1065	13.9
5.7	1.0225	3.2	12.2	1.0493	6.8	18.7	1.0775	10.35	25.2	1.1070	13.9
5.8	1.0229	3.2	12.3	1.0497	6.8	18.8	1.0779	10.4	25.3	1.1074	14.0
5.9	1.0233	3.3	12.4	1.0502	6.9	18.9	1.0783	10.5	25.4	1.1079	14.0
6.0	1.0237	3.3	12.5	1.0506	6.9	19.0	1.0788	10.5	25.5	1.1083	14.1
6.1	1.0241	3.4	12.6	1.0510	7.0	19.1	1.0792	10.6	25.6	1.1088	14.1
6.2	1.0245	3.4	12.7	1.0514	7.05	19.2	1.0797	10.6	25.7	1.1093	14.2
6.3	1.0249	3.5	12.8	1.0519	7.1	19.3	1.0801	10.7	25.8	1.1097	14.2
6.4	1.0253	3.6	12.9	1.0523	7.2	19.4	1.0806	10.7	25.9	1.1102	14.3
6.5	1.0257	3.6	13.0	1.0527	7.2	19.5	1.0810	10.8	26.0	1.1107	14.35

TABLE VI.—*Relation of brix, specific gravity, and Baumé—Continued.*

Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.
26.1	1.1111	14.4	32.6	1.1422	17.9	39.1	1.1748	21.4	45.6	1.2088	24.9
26.2	1.1116	14.5	32.7	1.1427	18.0	39.2	1.1753	21.5	45.7	1.2093	24.9
26.3	1.1121	14.5	32.8	1.1432	18.0	39.3	1.1758	21.5	45.8	1.2099	25.0
26.4	1.1125	14.6	32.9	1.1437	18.1	39.4	1.1763	21.6	45.9	1.2104	25.0
26.5	1.1130	14.6	33.0	1.1442	18.15	39.5	1.1768	21.6	46.0	1.2110	25.1
26.6	1.1135	14.7	33.1	1.1447	18.2	39.6	1.1773	21.7	46.1	1.2115	25.1
26.7	1.1140	14.7	33.2	1.1452	18.25	39.7	1.1778	21.7	46.2	1.2120	25.2
26.8	1.1144	14.8	33.3	1.1457	18.3	39.8	1.1784	21.8	46.3	1.2126	25.2
26.9	1.1149	14.8	33.4	1.1462	18.4	39.9	1.1789	21.85	46.4	1.2131	25.3
27.0	1.1154	14.9	33.5	1.1466	18.4	40.0	1.1794	21.9	46.5	1.2136	25.35
27.1	1.1158	14.9	33.6	1.1471	18.5	40.1	1.1799	22.0	46.6	1.2142	25.4
27.2	1.1163	15.0	33.7	1.1476	18.5	40.2	1.1804	22.0	46.7	1.2147	25.45
27.3	1.1168	15.1	33.8	1.1481	18.6	40.3	1.1809	22.1	46.8	1.2153	25.5
27.4	1.1172	15.1	33.9	1.1486	18.6	40.4	1.1815	22.1	46.9	1.2158	25.6
27.5	1.1177	15.2	34.0	1.1491	18.7	40.5	1.1820	22.2	47.0	1.2163	25.6
27.6	1.1182	15.2	34.1	1.1496	18.7	40.6	1.1825	22.2	47.1	1.2169	25.7
27.7	1.1187	15.3	34.2	1.1501	18.8	40.7	1.1830	22.3	47.2	1.2174	25.7
27.8	1.1191	15.3	34.3	1.1506	18.85	40.8	1.1835	22.3	47.3	1.2180	25.8
27.9	1.1196	15.4	34.4	1.1511	18.9	40.9	1.1840	22.4	47.4	1.2185	25.8
28.0	1.1201	15.4	34.5	1.1516	18.95	41.0	1.1846	22.4	47.5	1.2191	25.9
28.1	1.1206	15.5	34.6	1.1521	19.0	41.1	1.1851	22.5	47.6	1.2196	25.9
28.2	1.1210	15.55	34.7	1.1526	19.1	41.2	1.1856	22.5	47.7	1.2201	26.0
28.3	1.1215	15.6	34.8	1.1531	19.1	41.3	1.1861	22.6	47.8	1.2207	26.0
28.4	1.1220	15.7	34.9	1.1536	19.2	41.4	1.1866	22.65	47.9	1.2212	26.1
28.5	1.1225	15.7	35.0	1.1541	19.2	41.5	1.1872	22.7	48.0	1.2218	26.1
28.6	1.1229	15.8	35.1	1.1546	19.3	41.6	1.1877	22.75	48.1	1.2223	26.2
28.7	1.1234	15.8	35.2	1.1551	19.3	41.7	1.1882	22.8	48.2	1.2229	26.2
28.8	1.1239	15.9	35.3	1.1556	19.4	41.8	1.1887	22.9	48.3	1.2234	26.3
28.9	1.1244	15.9	35.4	1.1561	19.4	41.9	1.1892	22.9	48.4	1.2240	26.35
29.0	1.1248	16.0	35.5	1.1566	19.5	42.0	1.1898	23.0	48.5	1.2245	26.4
29.1	1.1253	16.0	35.6	1.1571	19.55	42.1	1.1903	23.0	48.6	1.2250	26.45
29.2	1.1258	16.1	35.7	1.1576	19.6	42.2	1.1908	23.1	48.7	1.2256	26.5
29.3	1.1263	16.1	35.8	1.1581	19.65	42.3	1.1913	23.1	48.8	1.2261	26.6
29.4	1.1267	16.2	35.9	1.1586	19.7	42.4	1.1919	23.2	48.9	1.2267	26.6
29.5	1.1272	16.25	36.0	1.1591	19.8	42.5	1.1924	23.2	49.0	1.2272	26.7
29.6	1.1277	16.3	36.1	1.1596	19.8	42.6	1.1929	23.3	49.1	1.2278	26.7
29.7	1.1282	16.4	36.2	1.1601	19.9	42.7	1.1934	23.3	49.2	1.2283	26.8
29.8	1.1287	16.4	36.3	1.1606	19.9	42.8	1.1940	23.4	49.3	1.2289	26.8
29.9	1.1291	16.5	36.4	1.1611	20.0	42.9	1.1945	23.45	49.4	1.2294	26.9
30.0	1.1296	16.5	36.5	1.1616	20.0	43.0	1.1950	23.5	49.5	1.2300	26.9
30.1	1.1301	16.6	36.6	1.1621	20.1	43.1	1.1955	23.55	49.6	1.2305	27.0
30.2	1.1306	16.6	36.7	1.1626	20.1	43.2	1.1961	23.6	49.7	1.2311	27.0
30.3	1.1311	16.7	36.8	1.1631	20.2	43.3	1.1966	23.7	49.8	1.2316	27.1
30.4	1.1315	16.7	36.9	1.1636	20.2	43.4	1.1971	23.7	49.9	1.2322	27.1
30.5	1.1320	16.8	37.0	1.1641	20.3	43.5	1.1976	23.8	50.0	1.2327	27.2
30.6	1.1325	16.85	37.1	1.1646	20.35	43.6	1.1982	23.8	50.1	1.2333	27.2
30.7	1.1330	16.9	37.2	1.1651	20.4	43.7	1.1987	23.9	50.2	1.2338	27.3
30.8	1.1335	17.0	37.3	1.1656	20.5	43.8	1.1992	23.9	50.3	1.2344	27.3
30.9	1.1340	17.0	37.4	1.1661	20.5	43.9	1.1998	24.0	50.4	1.2349	27.4
31.0	1.1344	17.1	37.5	1.1666	20.6	44.0	1.2003	24.0	50.5	1.2355	27.45
31.1	1.1349	17.1	37.6	1.1671	20.6	44.1	1.2008	24.1	50.6	1.2361	27.5
31.2	1.1354	17.2	37.7	1.1676	20.7	44.2	1.2013	24.1	50.7	1.2366	27.55
31.3	1.1359	17.2	37.8	1.1681	20.7	44.3	1.2019	24.2	50.8	1.2372	27.6
31.4	1.1364	17.3	37.9	1.1686	20.8	44.4	1.2024	24.2	50.9	1.2377	27.7
31.5	1.1369	17.3	38.0	1.1692	20.8	44.5	1.2029	24.3	51.0	1.2383	27.7
31.6	1.1374	17.4	38.1	1.1697	20.9	44.6	1.2035	24.35	51.1	1.2388	27.8
31.7	1.1378	17.4	38.2	1.1702	20.9	44.7	1.2040	24.4	51.2	1.2394	27.8
31.8	1.1383	17.5	38.3	1.1707	21.0	44.8	1.2045	24.45	51.3	1.2399	27.9
31.9	1.1388	17.55	38.4	1.1712	21.05	44.9	1.2051	24.5	51.4	1.2405	27.9
32.0	1.1393	17.6	38.5	1.1717	21.1	45.0	1.2056	24.6	51.5	1.2411	28.0
32.1	1.1398	17.7	38.6	1.1722	21.15	45.1	1.2061	24.6	51.6	1.2416	28.0
32.2	1.1403	17.7	38.7	1.1727	21.2	45.2	1.2067	24.7	51.7	1.2422	28.1
32.3	1.1408	17.8	38.8	1.1732	21.3	45.3	1.2072	24.7	51.8	1.2427	28.1
32.4	1.1412	17.8	38.9	1.1737	21.3	45.4	1.2077	24.8	51.9	1.2433	28.2
32.5	1.1417	17.9	39.0	1.1743	21.4	45.5	1.2083	24.8	52.0	1.2439	28.2

TABLE VI.—*Relation of brix, specific gravity, and Baumé—Continued.*

Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.
52.1	1.2444	28.3	58.6	1.2816	31.6	65.1	1.3205	34.95	71.6	1.3610	38.2
52.2	1.2450	28.3	58.7	1.2822	31.7	65.2	1.3211	35.0	71.7	1.3616	38.2
52.3	1.2455	28.4	58.8	1.2828	31.7	65.3	1.3217	35.05	71.8	1.3623	38.2
52.4	1.2461	28.4	58.9	1.2834	31.8	65.4	1.3223	35.1	71.9	1.3629	38.3
52.5	1.2467	28.5	59.0	1.2840	31.85	65.5	1.3229	35.15	72.0	1.3635	38.3
52.6	1.2472	28.5	59.1	1.2845	31.9	65.6	1.3235	35.2	72.1	1.3642	38.4
52.7	1.2478	28.6	59.2	1.2851	31.95	65.7	1.3241	35.25	72.2	1.3648	38.4
52.8	1.2483	28.65	59.3	1.2857	32.0	65.8	1.3247	35.3	72.3	1.3655	38.5
52.9	1.2489	28.7	59.4	1.2863	32.05	65.9	1.3253	35.35	72.4	1.3661	38.5
53.0	1.2495	28.75	59.5	1.2869	32.1	66.0	1.3260	35.4	72.5	1.3667	38.6
53.1	1.2500	28.8	59.6	1.2875	32.15	66.1	1.3266	35.4	72.6	1.3674	38.6
53.2	1.2506	28.85	59.7	1.2881	32.2	66.2	1.3272	35.5	72.7	1.3680	38.7
53.3	1.2512	28.9	59.8	1.2887	32.3	66.3	1.3278	35.5	72.8	1.3687	38.7
53.4	1.2517	28.9	59.9	1.2893	32.3	66.4	1.3285	35.6	72.9	1.3693	38.8
53.5	1.2523	29.0	60.0	1.2898	32.4	66.5	1.3291	35.6	73.0	1.3699	38.8
53.6	1.2529	29.1	60.1	1.2904	32.4	66.6	1.3297	35.7	73.1	1.3705	38.9
53.7	1.2534	29.1	60.2	1.2910	32.5	66.7	1.3303	35.7	73.2	1.3712	38.9
53.8	1.2540	29.2	60.3	1.2916	32.5	66.8	1.3309	35.8	73.3	1.3719	39.0
53.9	1.2546	29.2	60.4	1.2922	32.6	66.9	1.3315	35.8	73.4	1.3725	39.0
54.0	1.2551	29.3	60.5	1.2928	32.6	67.0	1.3322	35.9	73.5	1.3732	39.1
54.1	1.2557	29.3	60.6	1.2934	32.7	67.1	1.3327	35.9	73.6	1.3738	39.1
54.2	1.2563	29.4	60.7	1.2940	32.7	67.2	1.3334	36.0	73.7	1.3745	39.2
54.3	1.2568	29.4	60.8	1.2946	32.8	67.3	1.3340	36.0	73.8	1.3751	39.2
54.4	1.2574	29.5	60.9	1.2952	32.8	67.4	1.3346	36.1	73.9	1.3757	39.3
54.5	1.2580	29.5	61.0	1.2958	32.9	67.5	1.3352	36.1	74.0	1.3764	39.3
54.6	1.2585	29.6	61.1	1.2964	32.9	67.6	1.3359	36.2	74.1	1.3770	39.4
54.7	1.2591	29.6	61.2	1.2970	33.0	67.7	1.3365	36.2	74.2	1.3777	39.4
54.8	1.2597	29.7	61.3	1.2975	33.0	67.8	1.3371	36.3	74.3	1.3783	39.5
54.9	1.2602	29.7	61.4	1.2981	33.1	67.9	1.3377	36.3	74.4	1.3790	39.5
55.0	1.2608	29.8	61.5	1.2987	33.1	68.0	1.3384	36.4	74.5	1.3796	39.6
55.1	1.2614	29.8	61.6	1.2993	33.2	68.1	1.3390	36.4	74.6	1.3803	39.6
55.2	1.2620	29.9	61.7	1.2999	33.2	68.2	1.3396	36.5	74.7	1.3809	39.7
55.3	1.2625	29.9	61.8	1.3005	33.3	68.3	1.3402	36.5	74.8	1.3816	39.7
55.4	1.2631	30.0	61.9	1.3011	33.3	68.4	1.3408	36.6	74.9	1.3822	39.8
55.5	1.2637	30.05	62.0	1.3017	33.4	68.5	1.3415	36.6	75.0	1.3828	39.8
55.6	1.2642	30.1	62.1	1.3023	33.4	68.6	1.3421	36.7	75.1	1.3835	39.9
55.7	1.2648	30.15	62.2	1.3029	33.5	68.7	1.3427	36.7	75.2	1.3842	39.9
55.8	1.2654	30.2	62.3	1.3035	33.5	68.8	1.3433	36.8	75.3	1.3848	40.0
55.9	1.2660	30.25	62.4	1.3041	33.6	68.9	1.3440	36.8	75.4	1.3855	40.0
56.0	1.2665	30.3	62.5	1.3047	33.6	69.0	1.3446	36.9	75.5	1.3861	40.1
56.1	1.2671	30.4	62.6	1.3053	33.7	69.1	1.3452	36.9	75.6	1.3868	40.1
56.2	1.2677	30.4	62.7	1.3059	33.7	69.2	1.3458	37.0	75.7	1.3874	40.2
56.3	1.2683	30.5	62.8	1.3065	33.8	69.3	1.3465	37.0	75.8	1.3880	40.2
56.4	1.2688	30.5	62.9	1.3071	33.8	69.4	1.3471	37.1	75.9	1.3887	40.3
56.5	1.2694	30.6	63.0	1.3077	33.9	69.5	1.3477	37.1	76.0	1.3894	40.3
56.6	1.2700	30.6	63.1	1.3083	33.9	69.6	1.3484	37.2	76.1	1.3900	40.4
56.7	1.2706	30.7	63.2	1.3089	34.0	69.7	1.3490	37.2	76.2	1.3907	40.4
56.8	1.2712	30.7	63.3	1.3095	34.0	69.8	1.3496	37.3	76.3	1.3913	40.5
56.9	1.2717	30.8	63.4	1.3101	34.1	69.9	1.3502	37.3	76.4	1.3920	40.5
57.0	1.2723	30.8	63.5	1.3107	34.1	70.0	1.3509	37.4	76.5	1.3926	40.6
57.1	1.2729	30.9	63.6	1.3113	34.2	70.1	1.3515	37.4	76.6	1.3933	40.6
57.2	1.2735	30.9	63.7	1.3119	34.2	70.2	1.3521	37.5	76.7	1.3940	40.7
57.3	1.2740	31.0	63.8	1.3126	34.3	70.3	1.3528	37.5	76.8	1.3946	40.7
57.4	1.2746	31.0	63.9	1.3132	34.3	70.4	1.3534	37.6	76.9	1.3953	40.8
57.5	1.2752	31.1	64.0	1.3138	34.4	70.5	1.3540	37.6	77.0	1.3959	40.8
57.6	1.2758	31.1	64.1	1.3144	34.4	70.6	1.3546	37.7	77.1	1.3966	40.8
57.7	1.2764	31.2	64.2	1.3150	34.5	70.7	1.3553	37.7	77.2	1.3972	40.9
57.8	1.2769	31.2	64.3	1.3156	34.5	70.8	1.3559	37.8	77.3	1.3979	41.0
57.9	1.2775	31.3	64.4	1.3162	34.6	70.9	1.3565	37.8	77.4	1.3986	41.0
58.0	1.2781	31.3	64.5	1.3168	34.6	71.0	1.3572	37.9	77.5	1.3992	41.0
58.1	1.2787	31.4	64.6	1.3174	34.7	71.1	1.3578	37.9	77.6	1.3999	41.1
58.2	1.2793	31.4	64.7	1.3180	34.7	71.2	1.3585	38.0	77.7	1.4005	41.1
58.3	1.2799	31.5	64.8	1.3186	34.8	71.3	1.3591	38.0	77.8	1.4012	41.2
58.4	1.2804	31.5	64.9	1.3192	34.8	71.4	1.3597	38.1	77.9	1.4019	41.2
58.5	1.2810	31.6	65.0	1.3198	34.9	71.5	1.3604	38.1	78.0	1.4025	41.3

TABLE VI.—*Relation of brix, specific gravity, and Baumé—Continued.*

Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.
78.1	1.4032	41.3	80.1	1.4165	42.3	82.1	1.4300	43.3	84.1	1.4437	44.2
78.2	1.4039	41.4	80.2	1.4172	42.3	82.2	1.4307	43.3	84.2	1.4443	44.3
78.3	1.4045	41.4	80.3	1.4179	42.4	82.3	1.4314	43.4	84.3	1.4450	44.3
78.4	1.4052	41.5	80.4	1.4185	42.4	82.4	1.4320	43.4	84.4	1.4457	44.3
78.5	1.4058	41.5	80.5	1.4192	42.5	82.5	1.4327	43.5	84.5	1.4464	44.4
78.6	1.4065	41.6	80.6	1.4199	42.5	82.6	1.4334	43.5	84.6	1.4471	44.4
78.7	1.4072	41.6	80.7	1.4205	42.6	82.7	1.4341	43.5	84.7	1.4478	44.5
78.8	1.4078	41.7	80.8	1.4212	42.6	82.8	1.4348	43.6	84.8	1.4485	44.5
78.9	1.4085	41.7	80.9	1.4219	42.7	82.9	1.4354	43.6	84.9	1.4492	44.6
79.0	1.4092	41.8	81.0	1.4226	42.7	83.0	1.4361	43.7	85.0	1.4498	44.6
79.1	1.4098	41.8	81.1	1.4232	42.8	83.1	1.4368	43.7	85.1	1.4505	44.7
79.2	1.4105	41.9	81.2	1.4239	42.8	83.2	1.4375	43.8	85.2	1.4512	44.7
79.3	1.4112	41.9	81.3	1.4246	42.9	83.3	1.4382	43.8	85.3	1.4519	44.8
79.4	1.4119	42.0	81.4	1.4253	42.9	83.4	1.4388	43.9	85.4	1.4526	44.8
79.5	1.4125	42.0	81.5	1.4259	43.0	83.5	1.4395	43.9	85.5	1.4533	44.9
79.6	1.4132	42.1	81.6	1.4266	43.0	83.6	1.4402	44.0	85.6	1.4540	44.9
79.7	1.4138	42.1	81.7	1.4273	43.1	83.7	1.4409	44.0	85.7	1.4547	45.0
79.8	1.4145	42.2	81.8	1.4280	43.1	83.8	1.4416	44.1	85.8	1.4554	45.0
79.9	1.4152	42.2	81.9	1.4287	43.2	83.9	1.4423	44.1	85.9	1.4561	45.1
80.0	1.4158	42.2	82.0	1.4293	43.2	84.0	1.4430	44.2	86.0	1.4568	45.1

TABLE VII.—*Correction for the readings of Balling's saccharometer, on account of temperature.*

TO BE SUBTRACTED FROM THE DEGREE READ.

Temp.	Per cent of sugar in solution.													
C.	0	5	10	15	20	25	30	35	40	50	60	70	75	
13	0.14	0.18	0.19	0.21	0.22	0.24	0.26	0.27	0.28	0.29	0.33	0.35	0.39	
14	.12	.15	.16	.17	.18	.19	.21	.22	.22	.23	.26	.28	.32	
15	.09	.11	.12	.14	.14	.15	.16	.17	.16	.17	.19	.21	.25	
16	.06	.07	.08	.09	.10	.10	.11	.12	.12	.12	.14	.16	.18	
17	.02	.02	.03	.03	.03	.04	.04	.04	.04	.04	.05	.05	.06	
TO BE ADDED TO THE DEGREE READ.														
18	.02	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.02	
19	.06	.08	.08	.09	.09	.10	.10	.10	.10	.10	.10	.08	.06	
20	.11	.14	.15	.17	.17	.18	.18	.18	.19	.19	.18	.15	.11	
21	.16	.20	.22	.24	.24	.25	.25	.25	.26	.26	.25	.22	.18	
22	.21	.26	.29	.31	.31	.32	.32	.32	.33	.34	.32	.29	.25	
23	.27	.32	.35	.37	.38	.39	.39	.39	.40	.42	.39	.36	.33	
24	.32	.38	.41	.43	.44	.46	.46	.47	.47	.50	.46	.43	.40	
25	.37	.44	.47	.49	.51	.53	.54	.55	.55	.58	.54	.51	.48	
26	.43	.50	.54	.56	.58	.60	.61	.62	.62	.66	.62	.58	.55	
27	.49	.57	.61	.63	.65	.68	.68	.69	.70	.74	.70	.65	.62	
28	.56	.64	.68	.70	.72	.76	.76	.78	.78	.82	.78	.72	.70	
29	.63	.71	.75	.78	.79	.84	.84	.86	.86	.90	.86	.80	.78	
30	.70	.78	.82	.87	.87	.92	.92	.94	.94	.98	.94	.88	.86	

TABLE VIII.—*Allihn's table for the determination of dextrose.*

Milli-grams of cop-per.	Milli-grams of cu-prous oxid.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of cu-prous oxid.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of cu-prous oxid.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of cu-prous oxid.	Milli-grams of dex-trose.
11	12.4	6.6	76	85.6	38.8	141	158.7	71.8	206	231.9	105.8
12	13.5	7.1	77	86.7	39.3	142	159.9	72.3	207	233.0	106.3
13	14.6	7.6	78	87.8	39.8	143	161.0	72.9	208	234.2	106.8
14	15.8	8.1	79	88.9	40.3	144	162.1	73.4	209	235.3	107.4
15	16.9	8.6	80	90.1	40.8	145	163.2	73.9	210	236.4	107.9
16	18.0	9.0	81	91.2	41.3	146	164.4	74.4	211	237.6	108.4
17	19.1	9.5	82	92.3	41.8	147	165.5	74.9	212	238.7	109.0
18	20.3	10.0	83	93.4	42.3	148	166.6	75.5	213	239.8	109.5
19	21.4	10.5	84	94.6	42.8	149	167.7	76.0	214	240.9	110.0
20	22.5	11.0	85	95.7	43.4	150	168.9	76.5	215	242.1	110.6
21	23.6	11.5	86	96.8	43.9	151	170.0	77.0	216	243.2	111.1
22	24.8	12.0	87	97.9	44.4	152	171.1	77.5	217	244.3	111.6
23	25.9	12.5	88	99.1	44.9	153	172.3	78.1	218	245.4	112.1
24	27.0	13.0	89	100.2	45.4	154	173.4	78.6	219	246.6	112.7
25	28.1	13.5	90	101.3	45.9	155	174.5	79.1	220	247.7	113.2
26	29.3	14.0	91	102.4	46.4	156	175.6	79.6	221	248.7	113.7
27	30.4	14.5	92	103.6	46.9	157	176.8	80.1	222	249.9	114.3
28	31.5	15.0	93	104.7	47.4	158	177.9	80.7	223	251.0	114.8
29	32.7	15.5	94	105.8	47.9	159	179.0	81.2	224	252.4	115.3
30	33.8	16.0	95	107.0	48.4	160	180.1	81.7	225	253.3	115.9
31	34.9	16.5	96	108.1	48.9	161	181.3	82.2	226	254.4	116.4
32	36.0	17.0	97	109.2	49.4	162	182.4	82.7	227	255.6	116.9
33	37.2	17.5	98	110.3	49.9	163	183.5	83.3	228	256.7	117.4
34	38.3	18.0	99	111.5	50.4	164	184.6	83.8	229	257.8	118.0
35	39.4	18.5	100	112.6	50.9	165	185.8	84.3	230	258.9	118.5
36	40.5	18.9	101	113.7	51.4	166	186.9	84.8	231	260.1	119.0
37	41.7	19.4	102	114.8	51.9	167	188.0	85.3	232	261.2	119.6
38	42.8	19.9	103	116.0	52.4	168	189.1	85.9	233	262.3	120.1
39	43.9	20.4	104	117.1	52.9	169	190.3	86.4	234	263.4	120.7
40	45.0	20.9	105	118.2	53.5	170	191.4	86.9	235	264.6	121.2
41	46.2	21.4	106	119.3	54.0	171	192.5	87.4	236	265.7	121.7
42	47.3	21.9	107	120.5	54.5	172	193.6	87.9	237	266.8	122.3
43	48.4	22.4	108	121.6	55.0	173	194.8	88.5	238	268.0	122.8
44	49.5	22.9	109	122.7	55.5	174	195.9	89.0	239	269.1	123.4
45	50.7	23.4	110	123.8	56.0	175	197.0	89.5	240	270.2	123.9
46	51.8	23.9	111	125.0	56.5	176	198.1	90.0	241	271.3	124.4
47	52.9	24.4	112	126.1	57.0	177	199.3	90.5	242	272.5	125.0
48	54.0	24.9	113	127.2	57.5	178	200.4	91.1	243	273.6	125.5
49	55.2	25.4	114	128.3	58.0	179	201.5	91.6	244	274.7	126.0
50	56.3	25.9	115	129.6	58.6	180	202.6	92.1	245	275.8	126.6
51	57.4	26.4	116	130.6	59.1	181	203.8	92.6	246	277.0	127.1
52	58.5	26.9	117	131.7	59.6	182	204.9	93.1	247	278.1	127.6
53	59.7	27.4	118	132.8	60.1	183	206.0	93.7	248	279.2	128.1
54	60.8	27.9	119	134.0	60.6	184	207.1	94.2	249	280.3	128.7
55	61.9	28.4	120	135.1	61.1	185	208.3	94.7	250	281.5	129.2
56	63.0	28.8	121	136.2	61.6	186	209.4	95.2	251	282.6	129.7
57	64.2	29.3	122	137.4	62.1	187	210.5	95.7	252	283.7	130.3
58	65.3	29.8	123	138.5	62.6	188	211.7	96.3	253	284.8	130.8
59	66.4	30.3	124	139.6	63.1	189	212.8	96.8	254	286.0	131.4
60	67.6	30.8	125	140.7	63.7	190	213.9	97.3	255	287.1	131.9
61	68.7	31.3	126	141.9	64.2	191	215.0	97.8	256	288.2	132.4
62	69.8	31.8	127	143.0	64.7	192	216.2	98.4	257	289.3	133.0
63	70.9	32.3	128	144.1	65.2	193	217.3	98.9	258	290.5	133.5
64	72.1	32.8	129	145.2	65.7	194	218.4	99.4	259	291.6	134.1
65	73.2	33.3	130	146.4	66.2	195	219.5	100.0	260	292.7	134.6
66	74.3	33.8	131	147.5	66.7	196	220.7	100.5	261	293.8	135.1
67	75.4	34.3	132	148.6	67.2	197	221.8	101.0	262	295.0	135.7
68	76.6	34.8	133	149.7	67.7	198	222.9	101.5	263	296.1	136.2
69	77.7	35.3	134	150.9	68.2	199	224.0	102.0	264	297.2	136.8
70	78.8	35.8	135	152.0	68.8	200	225.2	102.6	265	298.3	137.3
71	79.9	36.3	136	153.1	69.3	201	226.3	103.1	266	299.5	137.8
72	81.1	36.8	137	154.2	69.8	202	227.4	103.7	267	300.6	138.4
73	82.2	37.3	138	155.4	70.3	203	228.5	104.2	268	301.7	138.9
74	83.3	37.8	139	156.5	70.8	204	229.7	104.7	269	302.8	139.5
75	84.4	38.30	140	157.6	71.3	205	230.8	105.3	270	304.0	140.0

TABLE VIII.—*Albim's table for the determination of dextrose—Continued.*

Milli-grams of cop-per.	Milli-grams of eu-prous oxid.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of eu-prous oxid.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of eu-prous oxid.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of eu-prous oxid.	Milli-grams of dex-trose.
271	305.1	140.6	321	361.4	168.1	371	417.7	196.3	421	474.0	225.1
272	306.2	141.1	322	362.5	168.6	372	418.8	196.8	422	475.6	225.7
273	307.3	141.7	323	363.7	169.2	373	420.0	197.4	423	476.2	226.3
274	308.5	142.2	324	364.8	169.7	374	421.1	198.0	424	477.4	226.9
275	309.6	142.8	325	365.9	170.3	375	422.2	198.6	425	478.5	227.5
276	310.7	143.3	326	367.0	170.9	376	423.3	199.1	426	479.6	228.0
277	311.9	143.9	327	368.2	171.4	377	424.5	199.7	427	480.7	228.6
278	313.0	144.4	328	369.3	172.0	378	425.6	200.3	428	481.9	229.2
279	314.1	145.0	329	370.4	172.5	379	426.7	200.8	429	483.0	229.8
280	315.2	145.5	330	371.5	173.1	380	427.8	201.4	430	484.1	230.4
281	316.4	146.1	331	372.7	173.7	381	429.0	202.0	431	485.3	231.0
282	317.5	146.6	332	373.8	174.2	382	430.1	202.5	432	486.4	231.6
283	318.6	147.2	333	374.9	174.8	383	431.2	203.1	433	487.5	232.2
284	319.7	147.7	334	376.0	175.3	384	432.3	203.7	434	488.6	232.8
285	320.9	148.3	335	377.2	175.9	385	433.5	204.3	435	489.7	233.4
286	322.0	148.8	336	378.3	176.5	386	434.6	204.8	436	490.9	233.9
287	323.1	149.4	337	379.4	177.0	387	435.7	205.4	437	492.0	234.5
288	324.2	149.9	338	380.5	177.6	388	436.8	206.0	438	493.1	235.1
289	325.4	150.5	339	381.7	178.1	389	438.0	206.5	439	494.3	235.7
290	326.5	151.0	340	382.8	178.7	390	439.1	207.1	440	495.4	236.3
291	327.4	151.6	341	383.9	179.3	391	440.2	207.7	441	496.5	236.9
292	328.7	152.1	342	385.0	179.8	392	441.3	208.3	442	497.6	237.5
293	329.9	152.7	343	386.2	180.4	393	442.4	208.8	443	498.8	238.1
294	331.0	153.2	344	387.3	180.9	394	443.6	209.4	444	499.9	238.7
295	332.1	153.8	345	388.4	181.5	395	444.7	210.0	445	501.0	239.3
296	333.3	154.3	346	389.6	182.1	396	445.9	210.6	446	502.1	239.8
297	334.4	154.9	347	390.7	182.6	397	447.0	211.2	447	503.2	240.4
298	335.5	155.4	348	391.8	183.2	398	448.1	211.7	448	504.4	241.0
299	336.6	156.0	349	392.9	183.7	399	449.2	212.3	449	505.5	241.6
300	337.8	156.5	350	394.0	184.3	400	450.3	212.9	450	506.6	242.2
301	338.9	157.1	351	395.2	184.9	401	451.5	213.5	451	507.8	242.8
302	340.0	157.6	352	396.3	185.4	402	452.6	214.1	452	508.9	243.4
303	341.1	158.2	353	397.4	186.0	403	453.7	214.6	453	510.0	244.0
304	342.3	158.7	354	398.6	186.6	404	454.8	215.2	454	511.1	244.6
305	343.4	159.3	355	399.7	187.2	405	456.0	215.8	455	512.3	245.2
306	344.5	159.8	356	400.8	187.7	406	457.1	216.4	456	513.4	245.7
307	345.6	160.4	357	401.9	188.3	407	458.2	217.0	457	514.5	246.3
308	346.8	160.9	358	403.1	188.9	408	459.4	217.5	458	515.6	246.9
309	347.9	161.5	359	404.2	189.4	409	460.5	218.1	459	516.8	247.5
310	349.0	162.0	360	405.3	190.0	410	461.6	218.7	460	517.9	248.1
311	350.1	162.6	361	406.4	190.6	411	462.7	219.3	461	519.0	248.7
312	351.3	163.1	362	407.6	191.1	412	463.8	219.9	462	520.1	249.3
313	352.4	163.7	363	408.7	191.7	413	465.0	220.4	463	521.3	249.9
314	353.5	164.2	364	409.8	192.3	414	466.1	221.0			
315	354.6	164.8	365	410.9	192.9	415	467.2	221.6			
316	355.8	165.3	366	412.1	193.4	416	468.4	222.2			
317	356.9	165.9	367	413.2	194.0	417	469.5	222.8			
318	358.0	166.4	368	414.3	194.6	418	470.6	223.3			
319	359.1	167.0	369	415.4	195.1	419	471.8	223.9			
320	360.3	167.5	370	416.6	195.7	420	472.9	224.5			

TABLE IX.—*Determination of maltose in beer.*

[According to Wein.]

Milli-grams of cop-per.	Milli-grams of eu-prous oxid.	Milli-grams of mal-tose.	Milli-grams of cop-per.	Milli-grams of eu-prous oxid.	Milli-grams of mal-tose.	Milli-grams of cop-per.	Milli-grams of eu-prous oxid.	Milli-grams of mal-tose.	Milli-grams of cop-per.	Milli-grams of eu-prous oxid.	Milli-grams of mal-tose.
31	34.9	26.1	36	40.5	30.5	41	46.2	34.8	46	51.8	39.1
32	36.0	27.0	37	41.7	31.3	42	47.3	35.7	47	52.9	40.0
33	37.2	27.9	38	42.8	32.2	43	48.4	36.5	48	54.0	40.9
34	38.3	28.7	39	43.9	33.1	44	49.5	37.4	49	55.2	41.8
35	39.4	29.6	40	45.0	33.9	45	50.7	38.3	50	56.3	42.6



TABLE IX.—*Determination of maltose in beer*—Continued.

Milli-grams of copper.	Milli-grams of cuprous oxid.	Milli-grams of maltose.	Milli-grams of copper.	Milli-grams of cuprous oxid.	Milli-grams of maltose.	Milli-grams of copper.	Milli-grams of cuprous oxid.	Milli-grams of maltose.	Milli-grams of copper.	Milli-grams of cuprous oxid.	Milli-grams of maltose.
51	57.4	43.5	116	130.6	100.8	181	203.8	159.2	246	277.0	217.2
52	58.5	44.4	117	131.7	101.7	182	204.9	160.1	247	278.1	218.1
53	59.7	45.2	118	132.8	102.6	183	206.0	160.9	248	279.2	219.0
54	60.8	46.1	119	134.0	103.5	184	207.1	161.8	249	280.3	219.9
55	61.9	47.0	120	135.1	104.4	185	208.3	162.7	250	281.5	220.8
56	63.0	47.8	121	136.2	105.3	186	209.4	163.6	251	282.6	221.7
57	64.2	48.7	122	137.4	106.2	187	210.5	164.5	252	283.7	222.6
58	65.3	49.6	123	138.5	107.1	188	211.7	165.4	253	284.8	223.5
59	66.4	50.4	124	139.6	108.0	189	212.8	166.3	254	286.0	224.4
60	67.6	51.3	125	140.7	108.9	190	213.9	167.2	255	287.1	225.3
61	68.7	52.2	126	141.9	109.8	191	215.0	168.1	256	288.2	226.2
62	69.8	53.1	127	143.0	110.7	192	216.2	169.0	257	289.3	227.1
63	70.9	53.9	128	144.1	111.6	193	217.3	169.8	258	290.5	228.0
64	72.1	54.8	129	145.2	112.5	194	218.4	170.7	259	291.6	228.9
65	73.2	55.7	130	146.4	113.4	195	219.5	171.6	260	292.7	229.8
66	74.3	56.6	131	147.5	114.3	196	220.7	172.5	261	293.8	230.7
67	75.4	57.4	132	148.6	115.2	197	221.8	173.4	262	295.0	231.6
68	76.6	58.3	133	149.7	116.1	198	222.9	174.3	263	296.1	232.5
69	77.7	59.2	134	150.9	117.0	199	224.0	175.2	264	297.2	233.4
70	78.8	60.1	135	152.0	117.9	200	225.2	176.1	265	298.3	234.3
71	79.9	61.0	136	153.1	118.8	201	226.3	177.0	266	299.5	235.2
72	81.1	61.8	137	154.2	119.7	202	227.4	177.9	267	300.6	236.1
73	82.2	62.7	138	155.4	120.6	203	228.5	178.7	268	301.7	237.0
74	83.3	63.6	139	156.5	121.5	204	229.7	179.6	269	302.8	237.9
75	84.4	64.5	140	157.6	122.4	205	230.8	180.5	270	304.0	238.8
76	85.6	65.4	141	158.7	123.3	206	231.9	181.4	271	305.1	239.7
77	86.7	66.2	142	159.9	124.2	207	233.0	182.3	272	306.2	240.6
78	87.8	67.1	143	161.0	125.1	208	234.2	183.2	273	307.3	241.5
79	88.9	68.0	144	162.1	126.0	209	235.3	184.1	274	308.5	242.4
80	90.1	68.9	145	163.2	126.9	210	236.4	185.0	275	309.6	243.3
81	91.2	69.7	146	164.4	127.8	211	237.6	185.9	276	310.7	244.2
82	92.3	70.6	147	165.5	128.7	212	238.7	186.8	277	311.9	245.1
83	93.4	71.5	148	166.6	129.6	213	239.8	187.7	278	313.0	246.0
84	94.6	72.4	149	167.7	130.5	214	240.9	188.6	279	314.1	246.9
85	95.7	73.2	150	168.9	131.4	215	242.1	189.5	280	315.2	247.8
86	96.8	74.1	151	170.0	132.3	216	243.2	190.4	281	316.4	248.7
87	97.9	75.0	152	171.1	133.2	217	244.3	191.2	282	317.5	249.6
88	99.1	75.9	153	172.3	134.1	218	245.4	192.1	283	318.6	250.4
89	100.2	76.8	154	173.4	135.0	219	246.6	193.0	284	319.7	251.3
90	101.3	77.7	155	174.5	135.9	220	247.7	193.9	285	320.9	252.2
91	102.4	78.6	156	175.6	136.8	221	248.7	194.8	286	322.0	253.1
92	103.6	79.5	157	176.8	137.7	222	249.9	195.7	287	323.1	254.0
93	104.7	80.3	158	177.9	138.6	223	251.0	196.6	288	324.2	254.9
94	105.8	81.2	159	179.0	139.5	224	252.4	197.5	289	325.4	255.8
95	107.0	82.1	160	180.1	140.4	225	253.3	198.4	290	326.5	256.6
96	108.1	83.0	161	181.3	141.3	226	254.4	199.3	291	327.4	257.5
97	109.2	83.9	162	182.4	142.2	227	255.6	200.2	292	328.7	258.4
98	110.3	84.8	163	183.5	143.1	228	256.7	201.1	293	329.9	259.3
99	111.5	85.7	164	184.6	144.0	229	257.8	202.0	294	331.0	260.2
100	112.6	86.6	165	185.8	144.9	230	258.9	202.9	295	332.1	261.1
101	113.7	87.5	166	186.9	145.8	231	260.1	203.8	296	333.2	262.0
102	114.8	88.4	167	188.0	146.7	232	261.2	204.7	297	334.4	262.8
103	116.0	89.2	168	189.1	147.6	233	262.3	205.6	298	335.5	263.7
104	117.1	90.1	169	190.3	148.5	234	263.4	206.5	299	336.6	264.6
105	118.2	91.0	170	191.4	149.4	235	264.6	207.4	300	337.8	265.5
106	119.3	91.9	171	192.5	150.3	236	265.7	208.3			
107	120.5	92.8	172	193.6	151.2	237	266.8	209.1			
108	121.6	93.7	173	194.8	152.0	238	268.0	210.0			
109	122.7	94.6	174	195.9	152.9	239	269.1	210.9			
110	123.8	95.5	175	197.0	153.8	240	270.2	211.8			
111	125.0	96.4	176	198.1	154.7	241	271.3	212.7			
112	126.1	97.3	177	199.3	155.6	242	272.5	213.6			
113	127.2	98.1	178	200.4	156.5	243	273.6	214.5			
114	128.3	99.0	179	201.5	157.4	244	274.7	215.4			
115	129.6	99.9	180	202.6	158.3	245	275.8	216.3			

TABLE X.—*Per cent of fat and solids not fat in milk.*

[According to Babcock.]

Per cent of fat.	Lactometer readings at 15.6° C.											Per cent of fat.
	26.	27.	28.	29.	30.	31.	32.	33.	34.	35.	36.	
0.0	6.50	6.75	7.00	7.25	7.50	7.75	8.00	8.25	8.50	8.75	9.00	0.0
0.1	6.52	6.77	7.02	7.27	7.52	7.77	8.02	8.27	8.52	8.77	9.02	0.1
0.2	6.54	6.79	7.04	7.29	7.54	7.79	8.04	8.29	8.54	8.79	9.04	0.2
0.3	6.56	6.81	7.06	7.31	7.56	7.81	8.06	8.31	8.56	8.81	9.06	0.3
0.4	6.58	6.83	7.08	7.33	7.58	7.83	8.08	8.33	8.58	8.83	9.08	0.4
0.5	6.60	6.85	7.10	7.35	7.60	7.85	8.10	8.35	8.60	8.85	9.10	0.5
0.6	6.62	6.87	7.12	7.37	7.62	7.87	8.12	8.37	8.62	8.87	9.12	0.6
0.7	6.64	6.89	7.14	7.39	7.64	7.89	8.14	8.39	8.64	8.89	9.14	0.7
0.8	6.66	6.91	7.16	7.41	7.66	7.91	8.16	8.41	8.66	8.91	9.16	0.8
0.9	6.68	6.93	7.18	7.43	7.68	7.93	8.18	8.43	8.68	8.93	9.18	0.9
1.0	6.70	6.95	7.20	7.45	7.70	7.95	8.20	8.45	8.70	8.95	9.20	1.0
1.1	6.72	6.97	7.22	7.47	7.72	7.97	8.22	8.47	8.72	8.97	9.22	1.1
1.2	6.74	6.99	7.26	7.49	7.74	7.99	8.24	8.49	8.74	8.99	9.24	1.2
1.3	6.76	7.01	7.24	7.51	7.76	8.01	8.26	8.51	8.76	9.01	9.26	1.3
1.4	6.78	7.03	7.28	7.53	7.78	8.03	8.28	8.53	8.78	9.03	9.28	1.4
1.5	6.80	7.05	7.30	7.55	7.80	8.05	8.30	8.55	8.80	9.05	9.30	1.5
1.6	6.82	7.07	7.32	7.57	7.82	8.07	8.32	8.57	8.82	9.07	9.32	1.6
1.7	6.84	7.09	7.34	7.59	7.84	8.09	8.34	8.59	8.84	9.09	9.34	1.7
1.8	6.86	7.11	7.36	7.61	7.86	8.11	8.36	8.61	8.86	9.11	9.37	1.8
1.9	6.88	7.13	7.38	7.63	7.88	8.13	8.38	8.63	8.88	9.13	9.39	1.9
2.0	6.90	7.15	7.40	7.65	7.90	8.15	8.40	8.66	8.91	9.16	9.41	2.0
2.1	6.92	7.17	7.42	7.67	7.92	8.17	8.42	8.68	8.93	9.18	9.43	2.1
2.2	6.94	7.19	7.44	7.69	7.94	8.19	8.44	8.70	8.95	9.20	9.45	2.2
2.3	6.96	7.21	7.46	7.71	7.96	8.21	8.46	8.72	8.97	9.22	9.47	2.3
2.4	6.98	7.23	7.48	7.73	7.98	8.23	8.48	8.74	8.99	9.24	9.49	2.4
2.5	7.00	7.25	7.50	7.75	8.00	8.25	8.50	8.76	9.01	9.26	9.51	2.5
2.6	7.02	7.27	7.52	7.77	8.02	8.27	8.52	8.78	9.03	9.28	9.53	2.6
2.7	7.04	7.29	7.54	7.79	8.04	8.29	8.54	8.80	9.05	9.30	9.55	2.7
2.8	7.06	7.31	7.56	7.81	8.06	8.31	8.57	8.82	9.07	9.32	9.57	2.8
2.9	7.08	7.33	7.58	7.83	8.08	8.33	8.59	8.84	9.09	9.34	9.59	2.9
3.0	7.10	7.35	7.60	7.85	8.36	8.61	8.10	8.86	9.11	9.36	9.61	3.0
3.1	7.12	7.37	7.62	7.87	8.38	8.63	8.13	8.88	9.13	9.38	9.64	3.1
3.2	7.14	7.39	7.64	7.89	8.40	8.65	8.15	8.90	9.15	9.41	9.66	3.2
3.3	7.16	7.41	7.66	7.92	8.42	8.67	8.17	8.92	9.18	9.43	9.68	3.3
3.4	7.18	7.43	7.69	7.94	8.44	8.69	8.19	8.94	9.20	9.45	9.70	3.4
3.5	7.20	7.45	7.71	7.96	8.46	8.71	8.21	8.96	9.22	9.47	9.72	3.5
3.6	7.22	7.48	7.73	7.98	8.48	8.73	8.23	8.98	9.24	9.49	9.74	3.6
3.7	7.24	7.50	7.75	8.00	8.50	8.75	8.25	9.00	9.26	9.51	9.76	3.7
3.8	7.26	7.52	7.77	8.02	8.52	8.77	8.27	9.02	9.28	9.53	9.78	3.8
3.9	7.28	7.54	7.79	8.04	8.54	8.79	8.29	9.04	9.30	9.55	9.80	3.9
4.0	7.30	7.56	7.81	8.06	8.56	8.81	8.31	9.06	9.32	9.57	9.83	4.0
4.1	7.32	7.58	7.83	8.08	8.58	8.83	8.33	9.08	9.34	9.59	9.85	4.1
4.2	7.34	7.60	7.85	8.10	8.60	8.85	8.35	9.11	9.36	9.62	9.87	4.2
4.3	7.36	7.62	7.87	8.12	8.62	8.88	8.37	9.13	9.38	9.64	9.89	4.3
4.4	7.38	7.64	7.89	8.14	8.64	8.90	8.39	9.15	9.40	9.66	9.91	4.4
4.5	7.40	7.66	7.91	8.16	8.66	8.92	8.41	9.17	9.42	9.68	9.93	4.5
4.6	7.43	7.68	7.93	8.18	8.68	8.94	8.43	9.19	9.44	9.70	9.95	4.6
4.7	7.45	7.70	7.95	8.20	8.70	8.96	8.45	9.21	9.46	9.72	9.97	4.7
4.8	7.47	7.72	7.97	8.22	8.72	8.98	8.47	9.23	9.48	9.74	9.99	4.8
4.9	7.49	7.74	7.99	8.24	8.74	9.00	8.49	9.25	9.50	9.76	10.01	4.9
5.0	7.51	7.76	8.01	8.26	8.76	9.02	8.51	9.27	9.52	9.78	10.03	5.0
5.1	7.53	7.78	8.03	8.28	8.79	9.04	8.53	9.29	9.54	9.80	10.05	5.1
5.2	7.55	7.80	8.05	8.30	8.81	9.06	8.55	9.31	9.56	9.82	10.07	5.2
5.3	7.57	7.82	8.07	8.32	8.83	9.08	8.57	9.33	9.58	9.84	10.09	5.3
5.4	7.59	7.84	8.09	8.34	8.85	9.10	8.60	9.36	9.61	9.86	10.11	5.4
5.5	7.61	7.86	8.11	8.36	8.87	9.12	8.62	9.38	9.63	9.88	10.13	5.5
5.6	7.63	7.88	8.13	8.39	8.89	9.15	8.64	9.40	9.65	9.90	10.15	5.6
5.7	7.65	7.90	8.15	8.41	8.91	9.17	8.66	9.42	9.67	9.92	10.17	5.7
5.8	7.67	7.92	8.17	8.43	8.94	9.19	8.68	9.44	9.69	9.94	10.19	5.8
5.9	7.69	7.94	8.20	8.45	8.96	9.21	8.70	9.46	9.71	9.96	10.22	5.9
6.0	7.71	7.96	8.22	8.47	8.98	9.23	8.72	9.48	9.73	9.98	10.24	6.0

TABLE XI.—Atomic weights. <sup>a</sup>

Name.	Sym- bol.	Atomic weight.		Name.	Sym- bol.	Atomic weight.	
		H=1.	O=16.			H=1.	O=16.
Aluminum.....	Al.....	26.9	27.1	Neodymium.....	.....	142.5	143.6
Antimony.....	St.....	119.5	120.4	Nickel.....	Ni.....	58.25	58.70
Arsenic.....	As.....	74.45	75.0	Nitrogen.....	N.....	13.93	14.04
Barium.....	Ba.....	136.4	137.40	Osmium.....	Os.....	189.6	191.0
Bismuth.....	Bi.....	206.5	208.1	Oxygen.....	O.....	15.88	16.000
Boron.....	B.....	10.9	11.0	Palladium.....	Pd.....	106.2	107.0
Bromine.....	Br.....	79.34	79.95	Phosphorus.....	P.....	30.75	31.0
Cadmium.....	Cd.....	111.55	112.4	Platinum.....	Pt.....	193.4	194.9
Cæsium.....	Cs.....	131.9	132.9	Potassium.....	K.....	38.82	39.1
Calcium.....	Ca.....	39.8	40.1	Præcodymium.....	.....	139.4	140.5
Carbon.....	C.....	11.9	12.0	Rhodium.....	Rh.....	102.2	103.0
Cerium.....	Ce.....	138.0	139.0	Rubidium.....	Rb.....	84.75	85.4
Chlorine.....	Cl.....	35.18	35.45	Ruthenium.....	Ru.....	100.9	101.7
Chromium.....	Cr.....	51.7	52.1	Samarium.....	Sm.....	149.2	150.3
Cobalt.....	Co.....	68.55	69.00	Scandium.....	Sc.....	43.8	44.1
Columbium.....	Cb.....	93.0	93.7	Selenium.....	Se.....	78.6	79.2
Copper.....	Cu.....	63.1	63.6	Silicon.....	Si.....	28.2	28.4
Erbium.....	Er.....	164.7	166.0	Silver.....	Ag.....	107.11	107.92
Fluorine.....	F.....	18.9	19.05	Sodium.....	Na.....	22.88	23.05
Gadolinium.....	.....	155.8	157.0	Strontium.....	Sr.....	86.95	87.60
Gallium.....	Ga.....	69.5	70.0	Sulphur.....	S.....	31.83	32.07
Germanium.....	Ge.....	71.9	72.5	Tantalum.....	Ta.....	181.5	182.8
Glucium.....	Gl.....	9.0	9.1	Tellurium.....	Te.....	126.5	127.52
Gold.....	Au.....	195.7	197.2	Terbium.....	Tb.....	158.8	160.00
Hydrogen.....	H.....	1.000	1.008	Thallium.....	Tl.....	202.61	204.15
Indium.....	In.....	113.1	114.0	Thorium.....	Th.....	230.8	232.6
Iodine.....	I.....	125.89	126.85	Thulium.....	Tu.....	169.4	170.7
Iridium.....	Ir.....	191.7	193.1	Tin.....	Sn.....	118.1	119.0
Iron.....	Fe.....	55.5	55.9	Titanium.....	Ti.....	47.8	48.15
Lanthanum.....	La.....	137.6	138.6	Tungsten.....	W.....	182.5	184.00
Lead.....	Pb.....	205.36	206.92	Uranium.....	U.....	237.8	239.6
Lithium.....	Li.....	6.97	7.03	Vanadium.....	V.....	51.0	51.4
Magnesium.....	Mg.....	24.1	24.3	Ytterbium.....	Yb.....	171.9	173.2
Manganese.....	Mn.....	54.6	55.0	Yttrium.....	Yt.....	88.3	89.0
Mercury.....	Hg.....	198.50	200.0	Zinc.....	Zn.....	64.9	65.4
Molybdenum.....	Mo.....	95.3	96.0	Zirconium.....	Zr.....	89.7	90.4

<sup>a</sup>Clarke, Jour. Am. Chem. Soc., 1901, 23, 90.



## APPENDIX.

As stated in the introduction, the methods given in the body of this report were submitted to about 250 chemists for criticism before they were reported to the association. The replies received were referred to the various authors and later were considered by them jointly at a meeting held on November 13, 14, 1901. All suggestions that those present approved of from their own experience were incorporated in the methods reported to the association and are published in this bulletin.

In this appendix are given extracts from replies containing other suggestions, which, though not adopted, were thought to be valuable and worthy of consideration at the hands of other analysts. At the beginning of each extract are indicated the page and the chapter subdivision of this bulletin containing the matter to which the criticism refers. The comments are by the referee.

### MEAT AND MEAT PRODUCTS.

Page 7, 1.—Practical men will sometimes say that this or that beef is cotton-seed-fed or slop-fed, judging simply from the appearance of the fat. No doubt such fats do give different factors which it might be well to take into account.—*B. M. Pillashy.*

Page 10, 4, 5, and 7 (a).—For the determination of water, ash, fat, and total nitrogen I have dried a weighed amount (50 grams) of finely chopped meat on a tared, flat, porcelain or nickel dish till the weight is approximately constant after it has stood in the atmosphere of the room over night. This is then immediately finely powdered and tightly stoppered, and is used for the above determinations.—*E. E. Smith.*

Page 10, 6.—Two grams of the dried sample seems an unnecessarily large quantity. I have used 0.5 to 1.0 gram. I think it would be well to specify "passed through a 100 (or 80) mesh sieve."—*E. E. Smith.*

Page 12, 7, (f).—It is quite common to determine meat bases directly by the bromin method without previous determinations of proteoses, peptones, and gelatin. How would the results thus obtained compare with those obtained according to (f)? Is the direct estimation of meat bases from the amount of nitrogenous matter not precipitated by bromin incorrect?—*A. P. Bryant.*

*Comment by Mr. Bigelow.*—Results by Mr. Trescot in this laboratory show a material error in the method suggested by Mr. Bryant, owing to the decomposition of meat bases with the evolution of nitrogen.

Page 16, 13.—I have succeeded best in extracting coloring matter from sausages by maceration with acidulated alcohol, using hydrochloric acid.—*A. S. Mitchell.*

### EDIBLE OILS AND FATS.

Page 20, 2.—In preference to the three methods given, I consider the Sprengel-tube method by far the most accurate, as well as the most rapid, process for determining the specific gravity at the boiling point. This is particularly the case when a large number of samples have to be examined together.

I may add that I consider accurate determinations of specific gravity especially

valuable when taken in conjunction with some other measurement (volatile acid, iodine, absorption, etc.). A fairly constant ratio may often be observed between the specific gravity and other measurements for a particular oil. Addition of an adulterant may upset this relationship without, however, bringing the specific gravity (or other measurement) outside the limits for the pure oil.—*Edgar B. Kenrick.*

Page 22, 2, (b), (2).—Why not use Westphal balance to determine specific gravity at temperature of boiling water?—*A. G. Woodman.*

Page 22, 3.—I think it is time that a vigorous protest be made against the practice, now becoming quite common, of reporting refractive indices by purely arbitrary numbers. One would think that the confusion resulting from the use of Twaddell, Baumé, etc., degrees of specific gravity should be sufficient warning.

The omission of any statement as to the optical relation between the readings of the Zeiss instrument and the refractive indices leaves the reader in doubt as to whether the table given is of any use beyond the individual instrument for which it was constructed. The writer of a recent text-book on physical measurements gives at the end of the book a table for the conversion of the readings of the Pulfrich refractometer into refractive indices. No warning is given to the reader that the table will not apply to any Pulfrich refractometer. The table, in fact, is quite useless for the instrument now in use in my laboratory. The index of refraction in the Pulfrich instrument is the square root of the difference between the square of the refractive index of the glass prism and the square of the sine of the angle measured. Since the refractive index of the prism in any particular instrument is not the same as that of the one for which the table in the book is made, it is obvious that the said table can not be used with my instrument.

After an experience of twelve years with two forms of the Pulfrich refractometer, I am able to say that this instrument leaves nothing to be desired in point of accuracy and ease of operation.—*Edgar B. Kenrick.*

Page 25, 4.—In weighing the fat for determination of iodine absorption I use Wesson's small, flat-bottomed glass cylinders and a narrow-mouthed bottle. (See our report for 1896, p. 23.) Would it not be well to make the instruction a little more elastic, so as to cover any form of glassware found efficient?—*A. L. Winton.*

Referring to the "wide-mouth bottle," I should prefer small glass stopper on account of greater danger of loss of iodine from large one.—*A. G. Woodman.*

Page 26, 5, (a).—It has always been customary in the laboratory at Munich, Bavaria, under Professor Hilger, to keep the solutions of iodine and mercuric chloride separate. They were mixed in equal proportions forty-eight hours before use.—*Emil Schlichting.*

Page 26, 5, (b) and (c).—Instead of about 5 grams of the melted fat, I think it better to use from 1 to 2 grams. Instead of a reflux condenser, I prefer a small funnel placed in mouth of flask. This is perfectly satisfactory and more convenient.—*A. G. Woodman.*

Page 29, 11.—The method of using 5 grams of fat is satisfactory for most analytical work, but that I may be able to present a sample of the adulterant found as evidence in court, in the case of adulterated linseed oils, it has been my practice to use 10 grams, following the method of Morawski and Demski, page 172 of Benedikt and Lewkowitch (Oils, Fats, and Waxes). By this method the removal of the alcohol used in saponification is avoided, as is also to a great extent the solubility of the alkaline soap solution in ether.

Ten grams of fat are saponified in a flask with 5 grams of caustic potash dissolved

in the least amount of water and 50 cc of stronger alcohol. The mixture is boiled for half an hour with inverted condenser. (I have found saponification by this method insufficient in the case of waxes, notably beeswax.) After saponification 50 cc of hot water are added, the mixture is cooled, and shaken out with petroleum ether.

Page 30, 12.—Commenting upon this method, I will state that while Wiley's method is undoubtedly the most accurate for determination of the melting point, the capillary-tube method is so much more convenient that most of the chemists in this section use it in their commercial work. I would suggest that that method be reinstated as an alternate method.—*A. S. Mitchell.*

Page 32, 14.—The directions for the Maumené figure are doubtless designed especially for olive oil; but inasmuch as cotton-seed, sesame, or poppy seed and the like might be included among edible oils, would it not be well to consider whether "strongest" sulphuric acid can be added to the latter oils without such frothing and evolution of  $\text{SO}_2$  as would vitiate the results? Some experiments on the Maumené test have recently been made in this laboratory\*. The details are now being prepared for publication.—*H. C. Sherman.*

*Comment by Mr. Tolman.*—This suggestion regarding the method for determining the Maumené figure is very appropriate and it will be necessary to modify the method when working with such oils as are mentioned above. Sherman\* uses an acid which will give a rise of temperature with water of  $33^\circ\text{--}34^\circ\text{C.}$ , and calculates the "specific temperature reaction" as given in the methods for oil analysis. But he finds that slightly lower results are obtained by this method. Each analyst should determine the constants for the conditions under which he is working.

Page 33, 17.—I should prefer to use the fatty acids for the Bechi test rather than the oil or fat itself.—*A. G. Woodman.*

#### DAIRY PRODUCTS.

Page 35, 2.—The dish used should be platinum or light porcelain. I should bar the use of either aluminum or tin. They are too easily subject to corrosion and change of weight. A small rod, about 1 inch by 3 or 4 millimeters, should be weighed with the dish, and the milk and sand should be stirred up when milk is added. This will greatly facilitate drying and no crust will form on top of the sand. In this manner more milk can be advantageously used, and I should advise increasing the amount to an optional 5 or even 10 cc. Anyone using the rod once will never after do without it.—*Charles L. Parsons.*

Page 36, 6.—From the expression, "if the milk contains one or more parts per 10,000 of formaldehyde," one would gather that the test will detect any amount of formaldehyde greater than one part per 10,000, whereas I believe that if the amount of formaldehyde be increased above a certain point the test will no longer show it.—*A. G. Woodman.*

Page 36, 6.—I think the test with phloroglucinol by far the easiest of use accompanied by certain identification, and should be given direct.—*Charles L. Parsons.*

Page 36, 6.—Under the detection of preservatives, I do not think it well to give the sulphuric acid test because there is such a variation between different chemists as to the reliability of this method and the manner of operating it. I would suggest the hydrochloric acid with ferric chlorid as more satisfactory and reliable, and its use is confined to the detection of formaldehyde in milk.

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\* "Science," 1901, p. 30.

Page 38, 7.—Under special tests for process butter I believe the appearance and qualitative tests of the curd are very valuable and should be given.<sup>a</sup>—*R. E. Doolittle.*

Page 38, 7.—In the examination of renovated butter by the Reichert process it has been my experience that where Leffman's glycerol method is used the results are uniformly lower than where saponification is effected with alcohol under pressure. It is also my experience that renovated butter when saponified, as is my habit, with alcohol, gives Reichert figures upon 5 grams of fat of from 26 to 28.5. Creamery butter, under these circumstances, gives slightly higher figures, ranging from 28 to 32 and very rarely higher. In other words, I find renovated butter to run upon an average two points lower than creamery butter.

Page 39, 9.—I am of the opinion that complete methods should be given for the detection of coloring matters in butter and its substitutes. This is one of the most important subjects that is engaging the attention of food chemists at the present time, because of the anticolor oleomargarine laws which have been enacted in the several States. I see the referee on Coloring Matter does not take up the subject of colors. In my work I make a preliminary test as follows: Take two portions of about 2 grams or more (according to apparent depth of color) of the filtered fat and dissolve each in a separate tube with ordinary ether. To one test tube add 1 or 2 cc. of dilute hydrochloric acid and to the other about the same quantity of dilute potassium hydroxid solution; shake both well and let stand. If the commonly used azo-dye be present, the first test tube will give a bright pink to deep wine-red color, according to amount of coloring matter present to the acid solution, while the potash solution of the second tube will be uncolored. On the other hand, if annatto be present the potash solution will be colored yellow, varying in depth with amount present, while the acid solution of the first tube will be uncolored. Having thus found the class the color probably belongs to, I can supply the different tests for that particular class.

For the azo color used in butter I would call attention to method of J. F. Geisler.<sup>b</sup> This is a very satisfactory and reliable method. For the vegetable colors, such as annatto, I think the methods of Martin and Cornwall should both be given complete.

Page 40, 3.—I would suggest that in my experience it has generally been sufficient to grind the cheese and place it in a muslin strainer upon the water bath for a short time, whereupon sufficient fat will be run out and may be filtered and dried. The addition of chemicals is thus wholly avoided.—*A. S. Mitchell.*

#### SPICES.

Page 55, 1.—To detect stems in ground cloves, shake up a little of the material in a test tube with alcohol (or other convenient solvent), allow the sediment to settle, and examine the absorption spectrum of the tincture for the characteristic bands of chlorophyll. It would appear that the green coloring matter is absent from the clove buds, while the stems contain it in considerable quantity.—*Edgar B. Kenrick.*

Page 55, 3.—I would recommend the determination of moisture (adventitious), as distinguished from volatile oil, by exposure in vacuo over colorless sulphuric acid. The moisture escapes before the volatile oil is appreciably volatilized. The marked discoloration of the acid may be taken as indicating the point at which notable quantities of volatile oil begin to come off. (See my work on cloves in Bul. 73.)—*A. McGill.*

*Comment by Mr. Winton.*—Certainly a promising method with obvious advantages, which should be studied by the association. Our standards are based, however, on analyses made by another method.

<sup>a</sup>Jour. Am. Chem. Soc., 1902, 22, 150.

<sup>b</sup>Jour. Am. Chem. Soc., 1898, 20, 110.



Page 55, 3.—I would suggest temperature of water bath, say about  $100^{\circ}$  instead of  $110^{\circ}$  C. A great many laboratories have only water baths, and the temperature obtainable is only  $100^{\circ}$  C.—*J. A. Le Clerc.*

*Comment by Mr. Winton.*—Temperature of  $110^{\circ}$ , although in some ways not so convenient as  $100^{\circ}$ , facilitates the removal of the volatile oils.

Page 56, 10.—Most published methods involve the assumption that oil of cloves exerts no vapor pressure at ordinary temperatures. This is very far from being the case. In the process described on page 2, a mixture of ether and oil of cloves will lose both ether and oil of cloves when allowed to "evaporate at room temperature." A second loss will take place in standing eighteen hours over sulphuric acid, the sulphuric acid continually absorbing the vapor of the oil as it is given off.

If cloves are finely powdered and exposed to the air in a thin layer, it will be found that after the lapse of a week or two the loss in weight which has taken place will be approximately equal to the amount of volatile oil originally present. The final weight, or rather the weight after the oil has evaporated, will vary slightly with the pressure of water vapor in the atmosphere. The fact that commercial ground cloves often contain very little volatile oil may sometimes be due to the fact that the cloves have been long ground, the oil having escaped by evaporation.

The correct method indicated would seem to be:

(1) Allow one of the volatile constituents to become in equilibrium with a limited atmosphere kept continually saturated with this constituent (e. g., water) at a given temperature.

(2) Allow the other volatile constituent (oil) to evaporate completely into an unlimited atmosphere free from this constituent.

(3) Restore the original conditions in (1).

The difference in weight between (1) and (3) will give the weight of the second constituent.

(All the oil and water may be driven off by a few hours' heating in a water oven.)

I have used various modifications of the above principle, but have so far had no opportunity of checking their absolute accuracy, as all published methods seem to me wrong in principle for the reason already given.—*E. B. Kenrick.*

Page 56, 10.—A student in this laboratory, Mr. L. L. Watters, last year made some experiments in regard to the determination of oil of sage by a method practically the same as this, except that very light petroleum ether was used for extraction. He found that it was difficult to drive off all of the ether without some of the oil, or to tell when all the solvent was driven off. Also sage oil left a residue, small, but rather variable, when evaporated, and brought to constant weight at  $100^{\circ}$  C. He thought the amount of residue thus left was influenced by the other constituents of the ether extract. A partial correction was obtained by adding to an aliquot part of the ether solution, before evaporation, a known weight of sage oil and carrying this through the same process as a blank. Possibly it might be worth while to try something of this sort with other volatile oils.—*H. C. Sherman.*

*Comment by Mr. Winton.*—Our standards are based on ether extraction. The method, we know, is not perfect, but we should go slow in making changes. The points named are worthy of further study.

Page 56, 10.—Could not petroleum ether or gasoline be used alternatively here or wherever extraction with absolute ether is recommended? The latter is better on account of the difficulty of keeping the ether perfectly anhydrous during the extraction.—*A. G. Woodman.*

*Comment by Mr. Winton.*—Petroleum ether has certain advantages over ether, but among the disadvantages in this instance is the fact that our standards of composition are based on the other method.

Page 56, 10.—Under determination of volatile and nonvolatile ether extracts I think he consumes too much time. He extracts with a continuous apparatus for twenty hours.—*J. A. Wesener.*

*Comment by Mr. Winton.*—The method for volatile and nonvolatile ether extract takes time, but I have found no satisfactory short cuts. Furthermore, our proposed standards are based on this method.

Page 56, 11.—Alcohol extracts from pepper a large amount (10 to 12 per cent) of matter which is easily volatile from its alcoholic solution, but is not volatile at the same temperature by dry heating. (See Bul. 10, Inland Revenue Laboratory, p, 18.)—*A. McGill.*

*Comment by Mr. Winton.*—A matter worthy of future study.

Page 57, 12.—The method we are using and one we much prefer to the copper-reducing method for sugars is to conduct the determination similarly to the copper-plating method until the copper suboxid has been dissolved in nitric acid. Then proceed as follows: Add sulphuric acid to displace the excess of nitric acid and boil until all fumes of nitric dioxid have disappeared. Then neutralize with sodium carbonate. Redissolve the precipitated copper in acetic acid, boil to expel excess of that acid, cool, and add enough potassium iodid to change all of the copper to cuprous iodid, with the liberation of free iodine. The free iodine thus liberated is then titrated with deci-normal ammonium thiosulphate, using starch as indicator. This method in our laboratory is both rapid and accurate.—*J. A. Wesener.*

*Comment by Mr. Winton.*—The association gives choice of several methods for determining of reduced copper, any of which may be employed. Personally, I prefer weighing either the  $\text{CuO}$  or  $\text{Cu}_2\text{O}$  in a Gooch crucible.

Page 57, 12.—In following Allihn's method, you direct that the solution, after addition of reducing solution, be heated merely to boiling, whereas in the A. O. A. C. version the solution is boiled two minutes.—*W. D. Bigelow.*

*Comment by Mr. Winton.*—The table used in connection with the Allihn method is based on Allihn's experiments with different quantities of pure dextrose, heating in each case until boiling begins again. So long as we use Allihn's table, we should follow his instructions. The writer, in a long series of experiments<sup>a</sup> with both pure starch and dextrose, found that the original Allihn method gave accurate results, whereas longer boiling brought the results too high. The German food analysts use the original Allihn method.

Page 57, 12.—We have found it better to make the copper solution very slightly acid. We include 1 cc of strong sulphuric acid in the 500 cc of copper sulphate solution.—*A. G. Woodman.*

*Comment by Mr. Winton.*—A subject for study by the association.

Page 58, 14.—Several inquiries have been received regarding the details of the method prescribed for the determination of crude fiber. Paper always gives a clear filtrate and nearly filters rapidly, whereas linen does not always retain the fine material and Gooch crucibles sometimes clog, rendering filtration impossible. A possible disadvantage of a weighed paper is that it may lose weight on treatment with soda, but this loss can be determined by blank experiment and a correction introduced. Of course a Gooch crucible is more convenient, provided it does not clog, but to make its use obligatory renders the method impossible for many samples.—*A. L. Winton.*

Page 58, 14.—In the determination of crude fiber, the centrifuge will be found a

<sup>a</sup> Conn. Expt. Sta. Rept., 1897, p. 128; Jour. Anal. Chem., 1888, p. 129.

great help; since filtration after use of alkali is almost impossible, and always most tedious.—*A. McGill.*

*Comment by Mr. Winton.*—Use of paper obviates the slow filtration.

Page 58, 14.—The only exception I take to the method of analysis as outlined is the method of estimating crude fiber. The official method is always tedious, and usually impossible with spices, owing to clogging of Gooch filter. The method as given is liable to be inaccurate, owing to difficulty of transferring fiber from paper, possibility of removing paper fiber, and the assumption that the weight of filter paper after washing with water, alcohol, and ether, and drying at 100 C.° is the same as before treatment. A method I have used with satisfaction consists in filtering through linen after acid, and again after alkali treatment, then transferring to Gooch crucible and proceeding as in official method.—*E. N. Eaton.*

*Comment.*—The method I recommend is that of the A. O. A. C., except that the fiber is weighed on a paper filter instead of the Gooch crucible. I can fully agree with Eaton that the "official method is always tedious and usually impossible with spices, owing to the clogging of Gooch filter," but I prefer a paper, both for the acid and alkali filtration. There is little danger of fiber being detached from the paper after the acid filtration.

Page 59, 16.—I think the Jena flasks used in determining nitrogen of nonvolatile ether extract should be described more exactly, whether round or flat-bottomed, length of neck, etc. Would it not be satisfactory to make extraction in the ordinary flask and transfer with water or ether? The flask you prescribe could not be used with the Knorr extraction apparatus.—*W. D. Bigelow.*

*Comment by Mr. Winton.*—I prefer a flat-bottomed Jena flask, 15 centimeters high, but other shapes and sizes may be used without affecting the result. The extract, after drying, is not easily removed with ether; hence it is strongly advised to digest for nitrogen in the extraction flask, transferring to a larger flask for distillation. The kind of apparatus used is not important, provided the extraction and digestion are complete, and mechanical loss is avoided.

#### FLAVORING EXTRACTS.

Page 70, 4.—I would suggest the following method for the estimation of vanillin:<sup>a</sup> Two cubic centimeters of the vanilla extract is measured into a test tube and sufficient freshly precipitated lead hydroxid added to completely decolorize. The mixture is washed onto a filter and the filtrate and washings collected in a Nessler tube. Bromin water is then added, after which enough of a freshly prepared 10 per cent solution of ferrous sulphate is added to get the maximum bluish-green color that will be produced if vanillin is present.

A standard solution is prepared by dissolving, say 50 mm of pure vanillin in 100 cc of water. A series of standards is then made, taking for instance  $\frac{1}{2}$ , 1, 2,  $2\frac{1}{2}$ , 3, etc., cc of the vanillin solution in Nessler tubes, each being treated with 2 or 3 drops of Bromin water, and with ferrous sulphate solution, and made up to the 50-cc mark.

The lead hydroxid is prepared by dissolving 200 grams of lead acetate in 850 cc of water. The solution is filtered and a strong solution of potassium hydroxid is added in excess, and the precipitate is washed thoroughly several times by decantation.<sup>b</sup>—*A. E. Leach.*

#### FERMENTED AND DISTILLED LIQUORS.

Page 83, 6.—When a liquor is titrated and the indicator does not give the color reaction distinctly on account of the color of the liquor after that point where the red goes to violet, the end reaction is gotten by dipping a capillary tube into the

<sup>a</sup> *Ztschr. anal. Chem.*, 1894, **33**, 242.

<sup>b</sup> *Ztschr. anal. Chem.*, 1894, **33**, 242; Mass. Board of Health Rept. for 1899, 629.

liquid after every other cubic centimeter and putting the point of it upon a piece of litmus paper. After the absorption of the liquor from the tube the center of the blot will show the alkaline blue on red litmus paper before a drop will. This method was brought before the local section of the American Chemical Society some years ago by Dr. S. Waldbott.—*B. M. Pilhashy.*

Page 84, 10.—Allow me to call attention to a point that is often overlooked in the examination of fermented and distilled liquors. The determination of glucose in wines, etc., is often based on the examination of the sample for dextrine. This in itself is all right except that very little commercial glucose is used in any fermented liquor except beer. The product that is used is grape sugar, which has been so highly converted that it will not give more than a qualitative test for dextrine. The only way to determine the presence of grape sugar is by double polarization before and after inversion. Nor is this positive proof of adulteration since, in some cases, invert sugar is used besides grape sugar, and the amounts are so well balanced that the polarization due to these two products is 0. In cases of this kind the addition of the foreign bodies can only be determined indirectly. The cane sugar is determined by reduction before and after inversion, the difference between the two results being calculated to cane sugar. The direct reduction is then due to the mixture of the grape and invert sugars. A mixture of about one part of grape sugar to to  $2\frac{1}{2}$  parts of invert sugar will give a direct polarization, and a polarization after inversion of 0. In some cases I have found that direct polarization was due to the cane sugar added. This is only the case where the fermentation was complete and all the sugar of the grape turned into alcohol.

The point that I want to bring out is that a certain relation exists between the amounts of sugars found by polarization before and after conversion, and the amounts of reducing sugars found by the reduction method before and after conversion, and that the absence of this relation is always proof of adulteration due to the addition of grape or invert sugar or both.—*Edward Gudeman.*

#### BAKING POWDER AND BAKING-POWDER CHEMICALS.

Page 98, 1.—Regarding the determination of carbon dioxid, I will say that I have used for factory control work a method which requires for its execution a slight modification of the Kjeldahl nitrogen apparatus, receiving the distillate in a solution of sodium hydroxid whose titre of free alkali is known; afterwards titrating with standard acid, using phenacetolin as the indicator. I am satisfied that with the cooperation of the association this method could be made as good for carbon dioxid as the Kjeldahl is for nitrogen.

Figure 7 illustrates the apparatus used by myself for the estimation of carbon dioxid in low-grade phosphate rock, such as is used for fillers in the manufacture of commercial fertilizers.

The flask C is an ordinary globe chemical flask having a capacity of 1,000 cc, fitted with a funnel tube having a stopcock at B. The flask E is an absorption flask having two bulbs, as shown. It should have a capacity of about 200 cc. The condenser D is a Liebig condenser of brass, with inner tube of block tin, as used in the Kjeldahl nitrogen process. This tin tube is connected with the bulb tube of flask C by a bit of rubber tubing and terminates in a glass tube which is fitted by a rubber stopper to flask E.

The determination is made as follows: Place 500 cc of freshly boiled distilled water in flask C; allow it to cool, and, if convenient, reduce its temperature to 20° or lower; then introduce into flask C about 1 gram of the substance to be estimated. Place in funnel tube a quantity of normal sulphuric acid considerably in excess of that required to decompose the substance. Place in the flask E 50 cc of normal or double normal sodium or potassium hydroxid solution. The exact content of hydroxid in this solution must be determined by titrating with decinormal sulphuric acid and phenacetolin previous to using it—in a separate portion, of course.

The apparatus is then connected, as shown in the illustration, with the glass tube dipping in the liquid in flask E. The stopcock B is then partly opened, and the acid is allowed to flow in at such a rate that the bubbles pass through E about one per second. Slowly increase the heat to boiling and distill about 50 cc.

After the distillation, titrate the liquid in E with decinormal sulphuric acid and phenacetolin, or, better, make it up to a definite quantity and take an aliquot part for titration, stopping at the first appearance of the pink. The difference between the amount of hydroxid shown by this last titration and that known to have been contained in it previous to receiving the distillate is the amount of hydroxid that has been converted into carbonate, and from this the quantity of carbon dioxid may readily be calculated. A check on the work may be made by titrating the yellow color, and the difference between the appearance of the pink and the appearance of the yellow is the amount of hydroxid in combination with carbon dioxid.

This method, for use in the determination of available carbon dioxid in baking powder, could be modified by placing the powder in C without water and introducing water gradually through the funnel tube and afterwards heating it as described. —*J. C. Mims.*

Page 100, 1, (a), (3).—It is convenient to introduce sample wrapped in a cartridge in tissue paper into the dry flask. The tissue paper may be colored with litmus or strips of litmus paper may be introduced at the same time. When the baking powder is badly made and contains excess of the acid ingredient this is indicated by the litmus remaining red at end of operation. Usually the sodium carbonate is in excess, and the paper remains blue.

Page 101, 1, (b), (3).—I find that 4 grams of a baking powder may safely and conveniently be employed in determining carbon dioxid. This reduces the factor needed to convert to percentage and conduces to accuracy. With a maximum value for baking powder this amount yields from 0.5 to 0.6 gram of carbon dioxid.

Page 104, 2.—I find it convenient to determine the excess of sodium carbonate occurring in most baking powders by replacing the tubes after determination of the available carbon dioxid, adding acid to the decomposing flask, and continuing the operation without recharging. The carbon dioxid now obtained is that due to excess of sodium bicarbonate. —*A. McGill.*

Page 104, 7.—We desire to make the following preliminary announcement of a method for the application of the polariscope to the estimation of tartaric acid in commercial products:

The commercial products containing tartaric acid fall into three classes, corresponding to the three methods of analysis described below:

*Class I.*—Tartaric acid and mixtures containing alkaline tartrates and calcium tartrate but no other optically active substances or materials capable of modifying the rotation of tartaric acid in ammoniacal solution (e. g., alum, iron). To this class belong Rochelle salt, potassium tartrate, cream of tartar, calcium tartrate, and many effervescing preparations of the Pharmacopœia.

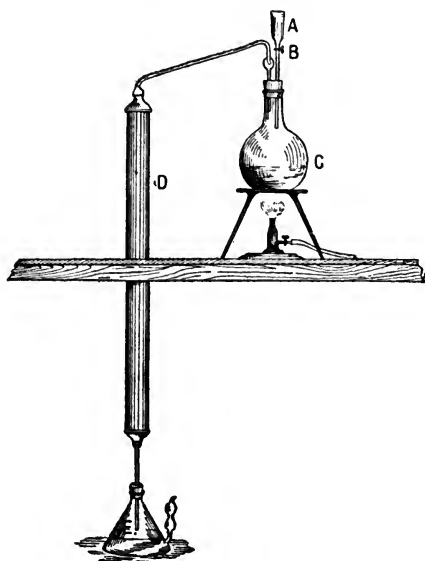


FIG. 7.—Mims' apparatus for the determination of carbon dioxid.

*Class II.*—Mixtures of the above (I) with sugar. To this class belong many of the effervescing compounds of the Pharmacopœia and most of the similar proprietary preparations.

*Class III.*—Mixtures of members of Class I with modifying agents and traces of optically active substances. To this class belong mixtures containing alum, those containing traces of iron and aluminum, and those of which starch is a constituent, the latter containing almost invariably traces of active substances soluble in cold water. Consequently all baking powders and mixtures of cream of tartar with cream of tartar substitutes fall into this division.

#### METHODS OF ANALYSIS.

*Class I.*—The method employed in the analysis of materials of this group is based on the fact that in the presence of excess of ammonia the rotation of the solution is proportional to the concentration of the tartaric acid, and is independent of the other bases and acids present.

(a) *The substance is completely soluble in dilute ammonia.*—A weighed quantity of the material containing not more than 1 gram tartaric acid is placed in a 25 cc measuring flask, moistened with 3 or 4 cc of water, and conc. ammonia (sp. gr. 0.880) added in quantity sufficient to neutralize all acids that may be present and leave about 1 cc in excess. The actual amount of the excess is not of importance, but a greater quantity than 1 cc of free ammonia should be avoided. The solution is then made up to 25 cc with water, filtered, if necessary, through a dry filter, and measured in a 20 cm tube in the polarimeter.

The amount of tartaric acid ( $C_4H_6O_6$ ) in grams ( $y$ ) in the material taken is given by the formula:

$$y = 0.00519x$$

where  $x$  is the rotation in minutes.

(b) *The substance is not completely soluble in dilute ammonia.*—In this case calcium tartrate is probably present and may be determined as follows: Treat 1 gram of the substance (or an amount containing not more than 1 gram tartaric acid) in a small beaker with 15 cc of water and 10 drops of conc. hydrochloric acid. Heat gently till both the potassium and calcium tartrates have passed into solution, and then, while still hot, add 2 cc of conc. ammonia (or enough to produce an ammoniacal smelling liquid) and about 0.1 gram of sodium phosphate dissolved in a little water. Transfer to a 25 cc measuring flask, cool, make up to the mark with water, filter through a dry filter, and polarize the filtrate in a 20 cm tube. The tartaric acid is calculated from the formula given under (a).

The precipitation of the calcium by means of sodium phosphate is not absolutely necessary, but when this is not done, in cases where the proportion of calcium in the sample is high, there is a great tendency for the calcium tartrate to crystallize out from the ammoniacal solution before the reading is made.

The tartaric acid present as bitartrate of potash may be determined by proceeding as in (a), the calcium tartrate being practically insoluble in cold ammonia solution.

The tartaric acid present as calcium tartrate is given, with sufficient accuracy for most purposes, by the difference between the results of (a) and (b). If more accurate results are required, the residue insoluble in ammonia in (a) may be dissolved in a little hydrochloric acid and treated as above with sodium phosphate and ammonia.

It may be noted that the method given below, under Class III, is applicable to this class also, but in most cases the above procedure will be found simpler.

*Class II.*—The determination of tartaric acid in substances of this class is an extension of the method given under I. In ammoniacal mixtures containing both tartaric acid and sugar the rotation of each is unaffected by the presence of the other substance, and consequently the rotation of the tartaric acid may be obtained by subtracting from the total rotation that part due to the sugar. The cane sugar may

be conveniently determined by Clerget's method, both readings (before and after inversion), however, being made after addition of ammonia. Should the sugar in these materials have become partly inverted, the reducing sugar must be determined by Fehling's process, and due allowance made for it.

*Class III.*—Direct readings of rotation in ammoniacal solution are inadmissible in analyses of the substances of this class on account of the influence of iron and aluminum on the rotation of tartaric acid, and on account of the small but unknown rotation of the trace of inverted starch.

Accurate determinations, however, may be made in the presence of excess of ammonium molybdate in neutral solution. The latter substance has the property of greatly increasing the rotation of tartaric acid, so that by its use the small rotation of the inverted starch is made insignificant. It is to be noted, however, that this increased rotation is very sensitive to the presence of alkali and acid, and is, moreover, modified by phosphates. It is therefore necessary, in the first place, to remove the phosphoric acid, and, secondly, to bring the solution to a definite state of neutrality. Both these results are attained by the following procedure, the details of which must be carefully adhered to:

*Solutions required.*—The following solutions must be prepared, but need not be made up very accurately:

Molybdate solution: 44 grams ammonium heptamolybdate in 250 cc.

Citric acid solution: 50 grams citric acid in 500 cc.

Magnesium sulphate solution: 60 grams  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  in 500 cc.

Ammonia solution: 80 cc concentrated ammonia (sp. gr. 0.880) in 500 cc.

Hydrochloric acid: 60 cc concentrated hydrochloric acid in 500 cc.

Methyl orange solution:

*Method of procedure.*—An amount of material containing not more than 0.2 gram tartaric acid, not more than 0.3 gram alum, and not more than 0.3 gram calcium superphosphate is accurately weighed and placed in a dry flask. To this, 5 cc. of citric acid and 10 cc. of molybdate solution are added and allowed to react with the substance for 10 or 15 minutes (with an occasional shake). Next, 5 cc. of magnesium sulphate solution are added and 15 cc. of ammonia solution stirred in. After a few minutes (not more than an hour) the solution is filtered through a dry filter, a slight turbidity of the filtrate being disregarded. To 20 cc. of the filtrate are then added a few drops of methyl orange and hydrochloric acid, from a burette, till the pink color appears (2 or 3 drops too much or too little are of no consequence). Finally, 10 cc. more of the molybdate solution are added to the pink solution, which now becomes colorless or pale yellow; and water is added to make up the volume to 50 cc. This solution, after filtering if necessary, is polarized in a 20-cm. tube.

The amount of tartaric acid in grams ( $y$ ) in the weight of substance originally taken is given by the following formula, in which  $x$  is the rotation in minutes:

$$y = 0.001086x + 0.001601 \sqrt{x}.$$

But if the rotation is not less than 40', the simpler formula,

$$y = 0.0075 + 0.001168x,$$

may be employed.

The following table gives the tartaric acid in grams for every 10 minutes rotation:

Rotation in minutes.	Grams tartaric acid.	Rotation in minutes.	Grams tartaric acid.
10.....	0.016	90.....	0.1130
20.....	0.029	100.....	0.1246
30.....	0.0415	110.....	0.1365
40.....	0.0535	120.....	0.1479
50.....	0.0657	130.....	0.1595
60.....	0.0776	140.....	0.1710
70.....	0.0895	150.....	0.1825
80.....	0.1013		

It may be mentioned that the temperature has practically no influence on the readings.—*Edgar B. Kenrick, University of Toronto; Frank B. Kenrick, University of Manitoba.*

Page 106, 12, (b).—Has anyone ascertained how accurately a mixed precipitate of ferric and aluminum phosphates can be calculated into its components from its gross weight and ( $\text{Al PO}_4=122$ ;  $\text{FePO}_4=151$ ;  $\text{P}_2\text{O}_5=142$ ) total phosphoric acid?

Let  $x$  and  $y=\text{Al PO}_4$  and  $\text{Fe PO}_4$ , respectively; then  $x+y=a$ ;

$$\text{and } \frac{142x}{244} + \frac{142y}{302} = b.$$

Page 107, 12, (d).—Would you not usually recommend the indirect calculation of K from the weight of mixed chlorids and total chlorin as being much less laborious than the precipitate of  $\text{K}_2\text{Pt Cl}_6$ ?—*A. McGill.*

#### FOOD PRESERVATIVES.

Page 108, 3.—It would be well to note that chloroform is much more convenient than ether for these extractions.

A more convenient way to apply the ferric chlorid test is to use a portion of the chloroform solution without evaporation. Shake this portion in a small test tube with about 1 cc of water, and add a very small drop of weak ferric chlorid solution; the violet color will be observed in the aqueous layer above the chloroform if salicylic acid is present. Successive small drops of ferric chlorid should be added until the deepest color is obtained or until it is evident that salicylic acid is not present. An excess of ferric chlorid must be avoided, as it tends to destroy the color; but, on the other hand, if the first addition of ferric chlorid does not produce the reaction, enough must be added to make sure that the color can not be obtained.

Page 109, 4.—The oxidation method devised by the writer (see Bulletin No. 59, U. S. Department of Agriculture, Division of Chemistry, page 60; or *Leffmann's Select Methods in Food Analysis*, 1901, page 98), based upon the oxidation of benzoic into salicylic acid (*Hanriot, C. R. 102, page 1250*), has been in use in this laboratory for nearly three years with good results, and I think it worthy a place in our methods. Recently Mr. J. O. LeBach, food chemist of this station, has simplified the manipulation by doing away with the cooling in ice water and performing the oxidation in a test tube instead of a dish. Mr. LeBach describes his method of making the test as follows:

Transfer about 0.05 gram to 0.1 gram of the dry residue from the chloroform or ether extract of the suspected sample to a test tube holding about 50 cc; add from 5 cc to 8 cc of concentrated  $\text{H}_2\text{SO}_4$ , shake until the mass is dissolved, then add from 0.5 to 0.8 gram of barium peroxid, a little at a time, shaking and cooling in water after each addition of the peroxid. After all the barium peroxid has been added a permanent froth should have been formed on the sulphuric acid. Let stand from 20 to 30 minutes, then fill the test tube three-fourths full of water, shake and cool rapidly to the temperature of the room. Filter off the barium sulphate and extract the filtrate with chloroform or ether. Draw off the chloroform and test it for salicylic acid in the usual way with dilute ferric chlorid.

It is important that during the oxidation of benzoic acid to salicylic acid with barium peroxid the temperature of the solution should not go higher than ordinary temperature; the cooler the better.

I have been able to oxidize benzoic acid to salicylic acid with commercial hydrogen peroxid, but have not found the method as easy to control as the one given above. Ammonium persulphate works as well in most cases, but sometimes fails to act properly. With barium peroxid I have never had a failure.

We find that saccharin, when oxidized in this manner, gives the salicylic acid reaction. It is therefore necessary, before applying this test, to determine by the taste whether saccharin is present. Of course it is necessary also first to prove the absence of salicylic acid.—*Alfred M. Peter.*



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